

SOVIET RESEARCH AND DEVELOPMENT IN MASS TRANSFER CHEMICAL ENGINEERING UNIT OPERATIONS




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TRANSFER CHEMICAL ENGINEERING UNIT OPERATIONS

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SOVIET RESEARCH AND DEVELOPMENT IN MASS TRANSFER CHEMICAL ENGINEERING UNIT OPERATIONS

INTRODUCTION

This report is concerned with Soviet capabilities in the chemical engineering unit operations of absorption (gas to liquid), adsorption (gas or liquid to solid), crystallization (liquid to solid) and extraction (liquid or solid to liquid). It is based on open Soviet technical literature of the period 1950-59.

The search for references and abstracts covered appropriate volumes of Chemical Abstracts, Monthly List of Russian Accessions (Library of Congress), Technical Translations (and its predecessor, Translations Monthly), Scientific Information Report, and the Chemistry Section of Referativnyi Zhurnal. The primary source was Chemical Abstracts, since a complete collection of this periodical was at hand; complete files of the others were available only for 1958 and 1959. The years 1955-59 were searched intensively and all appropriate Soviet papers noted; assuming a time lag of up to one year, this means the actual period of intensive search was 1954 through 1958. "Active" authors (arbitrarily defined as those with five or more publications in the 1955-59 period) were searched back to 1950 in Chemical Abstracts, and occasionally further to trace the development of significant research.

Discussion has been divided into four general sections, corresponding to the mass transfer operations surveyed. These are further subdivided as appropriate to the subject matter. An appendix contains tabulation of the principal Soviet authors, their latest known institutional affiliations, and their areas of specialty.

ABSORPTION

Soviet work on the theory and practice of absorption has been improving steadily. The best research in this area makes good use of modern mathematical and experimental techniques. Recent literature also indicates growing interest and capability in the development of industrial processes and equipment. The following discussion is divided into theoretical and practical considerations.

Theoretical Considerations

An extensive series of articles of good quality has been published by M. Kh. Kishinevskiy and his co-workers in the field of absorption processes. These range from studies of practical significance to those which are highly theoretical. Many of the studies make intensive use of the knowledge of techniques and practices of the Western world. Emphasis frequently is given to absorption in systems where intensive mixing is encountered. For example, Kishinevskiy, A. V. Pamfilov, and M. A. Kerdivarenko have studied the absorption of carbon dioxide by solutions of sodium hydroxide and sodium carbonate

under high-speed agitation. 1/2/ A closed system was used, and rate of absorption was determined by recording decrease of pressure in the vapor phase. They developed a general equation for the kinetics of the absorption process, which gave the parameter modifications as functions of the degree of turbulence in the reacting phase. 3/

Consideration of mass flow transfer occurring across a surface boundary during absorption was used by Kishinevskiy to develop a general equation that is valid for first-order reactions and holds over a wide range of turbulence. Where $\frac{dw}{6\Delta T}$ is the flow of material across the boundary per unit time, he has shown that:

$$\frac{dw}{6\Delta T} = \frac{MC \left[1 - e^{-\left(P - \frac{dw}{6\Delta T} \times \frac{1}{\beta} \right) HK_C \Delta T} \right]}{\frac{1}{V_n} + \frac{H}{\beta}} + HP$$

where, β and V_n are hydrodynamic constants for the gas and liquid phase, respectively, giving the linear flow normal to the surface; C is the concentration of the absorbate in the absorbent; P is partial pressure in the gas phase; H is Henry's law constant; M is a stoichiometric constant for the reaction; and K_C is a constant proportional to the reaction-velocity constant. 4/ This was further discussed in relation to the transfer of a substance in the gaseous phase during absorption by a liquid absorbent. 5/

Working with S. M. Fayer and R. M. Novik, respectively, Kishinevskiy determined and presented experimental data for sulfur dioxide absorbed by potassium hydroxide and for carbon dioxide absorbed by sodium carbonate. 6/ 7/ The apparatus used was a closed system with vigorous agitation. The rate of absorption was shown to follow the equation deduced theoretically from the kinetics of the process.

It was demonstrated experimentally by Kishinevskiy that molecular diffusion plays practically no part in the transfer of material from the free surface into the volume under conditions of high turbulence for the absorption of hydrogen, nitrogen and oxygen in distilled water. The decisive factor in this process was turbulent diffusion. The data were correlated by means of a theoretical equation:

$$W = VC^* (1 - e^{-K_1 T/V})$$

where W is the quantity of gas absorbed, V is the volume of absorbing liquid, C^* is the surface concentration of the absorbed gas, and T is the time. Kishinevskiy based this theory and equation on a concept of surface layer renewal for absorption processes. This excellent concept was formulated by Kishinevskiy prior to its publication by other investigators. 8/

According to Kishinevskiy, the differential equations previously derived for absorption processes accompanied by second-order chemical reactions (chemical absorption) can be solved, assuming that there is no need for a correlation between the concentration at the interface surface, the concentration at a distance beneath the surface, and the "age" of the surface

as a function of turbulence. 9/

Kishinevskiy and L. A. Mochalova developed a theory of absorption in a process wherein some chemical reaction takes place, e.g., carbon dioxide in water. Contrary to the accepted two-film theory of Lewis and Whitman, they consider absorption as a quasi-stationary process during which there is a periodic renewal of the surface, i.e., the interface between gas and liquid. 10/ This relatively new concept permits good theoretical correlation of absorption data.

The rates of absorption of carbon dioxide in sodium hydroxide, sodium carbonate, and monoethanolamine solutions, and of sulfur dioxide in sodium carbonate solution were determined. Temperature and concentration were adjusted to give a constant viscosity for formation of constant gas bubble sizes during absorption. The results show the kinetics can be analyzed by simplified equations dependent on the solubilities of the systems. 11/

Previous work by other investigators had indicated that very little ethylene is absorbed by sulfuric acid, especially when the acid has been diluted by water. M. A. Kerdivarenko, P. K. Migal and Kishinevskiy demonstrated that it is possible to describe quantitatively the rate of absorption of ethylene by aqueous sulfuric acid solutions, using the previously deduced equations based on the renewal of surface layers by intensive turbulent stirring. 12/

Traditionally, the approach to the theoretical analysis of gas absorption by liquids consists of the quantitative determination of the rate of diffusion by means of an equation:

$$q = \frac{D}{d} (C_0 - C)$$

in which q is the mass flow; D is the coefficient of molecular diffusion; d is the thickness of the diffusion layer; and C_0 and C are the mass concentrations at the margins of the diffusion layer. This equation is a mathematical expression of two physical hypotheses: (1) the existence of a sublayer of diffusion, and (2) the stationary nature of the process. Kishinevskiy denied the adequacy of the traditional approach of the so-called two-film absorption scheme and considered the contact between the gas and liquid interacting phases of brief duration. This led to the following equation:

$$q = - D_T \frac{\partial C}{\partial y}$$

in which, $\partial C / \partial y$ is the concentration gradient; and D_T is the effective coefficient of diffusion, including both turbulent and molecular diffusion. Further development of this second sound approach will permit theoretical analysis of absorption processes that are complicated by chemical reactions. 13/

The absorption of carbon dioxide by sodium hydroxide solutions was studied by Kishinevskiy and Mochalova to further develop the theory of the quasi-stationary concept in absorption processes when accompanied by chemical

reactions. Their experimental data showed the contact time, ΔT , between the liquid and gas phases, i.e., "bubbles", could be calculated by the simple equation: $\Delta T = \frac{r}{w}$ in which, r is the bubble radius; and w is the rate

of ascent of the bubble of gas through the liquid. Further, the value of the effective coefficient of diffusion, D_T , was much greater than the traditional coefficient of molecular diffusion, D , leading to much greater absorption.

14/

Kishinevskiy continued his work on the development of the basic equation for absorption processes accompanied by second-order chemical reactions. Experimental data were determined and used to confirm the adequacy of theoretical equation for carbon dioxide absorbed in solutions of potassium hydroxide. With high stirring velocity, i.e., turbulent absorption, there was no gas film resistance. 15/

Kishinevskiy and Mochalova developed and experimentally verified a method for determining the requisite design height of packed absorption columns when a chemical reaction occurs between absorbent and absorbate. The equation derived assumed the concentration of the absorbent was constant along the column. This assumption is true only at relatively high liquor rates and low gas velocities. 16/

Based on the reported studies and theoretical developments, good methods for designing absorption towers for gas-liquid interactions, i.e., chemical absorption, had not been adequately developed. A method was proposed by Mochalova and Kishinevskiy for calculating the height of absorption towers. By use of this method, equipment can be designed to absorb a component from the gaseous mixture to a prescribed degree and to give a liquid of a definite concentration. 17/

M. Ye. Pozin and his several co-workers have carried out a series of studies on absorption with chemical reaction, i. e., chemical absorption, to develop a theory of such a process. Some of the systems investigated were sulfur dioxide and sodium carbonate; ammonia and sulfuric acid; and carbon dioxide and potassium hydroxide. 18-20/ With Ye. S. Tumarkina, Pozin also studied bromine and sodium hydroxide; bromine and sodium carbonate; bromine and ferrous bromide; and bromine and ferrous bromide. 21/

Whenever the chemical reaction of a gas with a solution is reversible and attains equilibrium before stoichiometric completion, the chemical capacity of the absorbent is not reached but the effective concentration of the absorbent acts apparently smaller. Equations were developed by Pozin to explain these phenomena. 22/ He also derived equations to explain gas absorption with crossed flows of gas and liquid by assuming that the concentration of the absorbed component in the gas varies linearly with the length of the path. The derived equations are standard and advance no new concepts. 23/

Pozin, I. P. Mukhlenov and Ye. Ya. Tarat found the conditions of absorption in a sieve-plate apparatus are determined primarily by the flow rate of the gas stream. This bubbling process then passes quickly

into a foaming process, wherein mass transfer proceeds more vigorously in the layers of dynamically fluidized foam consisting of films and liquid mixed with the gas bubbled. 24/ Pozin is continuing his studies of absorption under foaming conditions, having reported recently with B. A. Kopylev and N.A. Petrova on the efficiency of a four-plate apparatus for ammonia absorption in copper-ammonia solution. 25/ This work, though incomplete, is promising.

In the extensive literature on absorption of gases by liquids, attention has been devoted to rates of the process while relatively little emphasis has been given to the degree of reaction of the absorption process components. Pozin and B. A. Kopylev stated that the efficiency of absorption depends on both the rate and the degree of inter-reaction. An approximate equation, derived for crossflow of a gas and liquid, established the relationship between absorption efficiency, absorption coefficient, and linear gas velocity:

$$n = \frac{2K}{2W + K}$$

where, n is the fractional efficiency of the plate (or tray) with respect to the gas; K is the total absorption coefficient; and, W is the linear velocity of the inert component of the gas stream. Using this equation and experimental data, they proved that the absorption efficiency in bubbling and foam equipment was determined mainly by the solubility of the gas in the liquid, while other factors (linear gas and liquid velocities, height of the bubbling or foam layer, etc.) varied the efficiency only within definite limits. 26/

To demonstrate their chemical absorption theory Pozin and his associates also used other systems of interest: nitrogen oxides and calcium hypochlorite; hydrogen sulfide and arsenate-carbonate solutions. 27/ 28/ These articles are good and emphasize the importance of these new penetration concepts.

V. V. Kafarov has analyzed several sets of data on the absorption of carbon tetrachloride by benzene. His analysis led him to conclude that free turbulence approaching emulsification, i.e., the range of turbulence where the Reynolds Number is independent of the Peclet Group, was essential for the scaling up of two-phase systems. 29/ In a continuing analysis and review, Kafarov and Yu. I. Dytnerkiy arrived at a similar conclusion. 30/

The two-film theory for mass transfer processes was criticized by Kafarov because "the film theory ignores the hydrodynamic conditions process, as strictly defined conditions are laid down -- the presence of molecular diffusion, which, as is known, can only be significant in stationary layers or in layers moving at low velocities." With the development of free turbulence and the formation of eddies at the interface, conditions are set up, depending on the hydrodynamics, in which the coefficients of turbulent diffusion may increase to a point where molecular film transfer may be neglected. In absorption processes (with moving two-phase streams), the mass transfer was expressed by him as:

$$q = -(D + E_g) \frac{dC}{dx}$$

in which, q is the mass transferred; D is the molecular diffusion; E_g is

the turbulent transfer component; dC is the concentration change over a transfer distance dx . If one defines: $K = D + E_g$, then the former equation is reduced to: $q = K \Delta C / L$, in which K represents both the molecular and turbulent mass transfer over the finite length, L . In order to apply these generalizations to an analysis of diffusion processes, Kafarov stated that the determining criterion for mass transfer should be one for turbulent mass transfer, i.e., a modified Peclet number. 31/ This theory was further expanded by Kafarov to an analysis by use of similitude principles so that the experimental results could be transferred to the design of industrial processes. 32/ 33/

Kafarov and V. I. Trofimov have analyzed a series of experiments on absorption of ammonia, sulfur dioxide, acetone, ethanol and carbon dioxide by water in packed towers. They conclude that free turbulence begins at an "inversion point," where the gas passes from the continuous phase to the dispersed phase and liquid passes from the dispersed phase to the continuous phase. 34/ 35/

Consideration of these analyses and theories was developed by Kafarov and L. I. Blyakham into a Soviet patent. The most valuable claim can be stated as follows: Conditions are created which cause emulsification within the packed column. The yields per unit volume for absorption, rectification and heat or mass exchange are increased by this method. 36/

L. D. Berman also expostulated against the use of the traditional, stationary two-film concept in analyzing mass transfer processes, i.e., absorption processes. Speaking of the total resistance to mass transfer as equal to the sum of the partial resistances of the liquid and gas phases, he stated: "Such a division, which has an adequate physical basis, of the total resistance into its components does not require the introduction of a concept of fictitious gas and liquid films. ...This concept of equivalent or derived films, which has penetrated into our literature from foreign sources, presents no advantages, but is harmful because it distracts attention from a correct evaluation of the actual hydrodynamic conditions and in consequence it sometimes leads to erroneous conclusions." 37/ Even though the Soviets object to the "two-film" concept, valuable correlations have resulted from its use.

Many investigators have stressed the importance of the similarity of mass exchange and heat transfer, assuming a fixed phase interface. Berman pointed out the applicability of this similarity in some practical cases but warned that the peculiarities in mass exchange (such as transverse flow of components, molar flow of substance, and turbulence in the interface layer) destroy much of the similarity between the mass exchange process and the heat transfer process. He confirmed this new criterion equation by the use of experimental data, developed by him and others. 38/ This equation is important.

The process of absorption with chemical reaction was carefully described by A. M. Rozen, who used it to develop a method of calculating isotope exchange in the following reactions:



His explanation was intriguing, and his study has potential valuable implications. 39/

In absorption processes, wherein mass transfer takes place between a soluble absorbate in a cavity of a gas bubble and the liquid around it, hydrodynamic considerations govern the shape and dimensions of the bubbles. Ladyzhenskiy presented an excellent mathematical discussion of this phenomenon that is important in "bubbling" absorption. From this study he concluded that the shape of a spheroidal bubble ascending in a still liquid is determined mainly by gravitational forces. 40/

A good mathematical development of kinetic equations for the absorption process, when reversible chemical reactions take place in the absorbent, was presented by I. G. Plit. 41/ He considered the kinetics of absorption of carbon dioxide by potassium carbonate from the above point of view as represented by a film consisting of neutralized and non-neutralized zones. 42/

Earlier studies by Plit, working with K. N. Shishkin, covered the absorption of carbon dioxide by a bubble of foam rising from a potassium carbonate solution. Their results showed that an increase of foam agent (saponin) concentration increased the absorption rate of carbon dioxide by the solution. 43/ Plit also described very clearly the kinetics of "stripping" (desorption) of hydrogen sulfide from the absorbent (potassium carbonate and potassium bicarbonate solution). The rate of desorption fell rapidly after the initial stripping period but increased as the total heat input was increased. 44/ 45/

Further evidence was advanced by V. M. Ramm and A. Yu. Zakgeym to show that the two-film theory of absorption can not be corrected or proved. They stated that the non-stationary diffusion theory, which assumes that fresh liquid moves to the interface and removes liquid that has already interacted with the gas, is more exact. 46/

Calculations were suggested for design characteristics of packed (spray type) and of bubble plate (sectional type) absorption equipment by V. M. Govorkov and Ya. D. Averbukh. They emphasized that, in the first type, there is a continuous and monotonous change in the driving force; and, in the second type, there is a "step" change in the driving force. Therefore, in the latter type, the design must be calculated by graphical methods. 47/

A relationship was established between the mass transfer coefficient in the gaseous phase and the volumetric velocity of the gas and the depth of immersion by G. N. Gasyuk, et al. 48/ In general, it can be stated that the Soviet theoretical work is good, even though it is argumentative on the value of the "two-film" concept.

Practical Considerations

Many articles of practical significance have been published in recent Soviet literature. Of these, the studies of S. N. Ganz and his co-workers on nitrogen oxide absorption for production of nitric acid and for recovery of nitrogen oxides used in the lead chamber production of sulfuric acid are the most extensive and important.

Recovery by absorption of nitric oxide in ferrous sulfate solutions was investigated by Ganz and L. I. Mamon over a wide range of operating conditions: nitric oxide concentrations in gas and solution; ferrous sulfate concentration; temperature; gas flow rate; and solution rate. Optimum conditions were determined for efficient recovery. 49/

With S. B. Kravchinskaya, Ganz studied the absorption rate of nitrogen oxides in solutions of calcium hydroxide in a revolving plate absorber. The rate increased rapidly with the peripheral speed of the plates. They calculated that the construction cost and operating energy of a revolving absorber are 10 percent and 38.4 percent of an equivalent capacity packed tower. 50/

The rate and completeness of the conversion of nitrogen oxides into nitric acid depends upon a number of factors including temperature, the concentration of nitrogen oxides in the gas, degree of oxidation, concentration of absorbing acid, and liquid and gas rates. This multiplicity of factors makes the kinetics of nitric acid formation a very complex process. Ganz, M. A. Lokshin, and S. I. Kapturova developed equations that account for the effect of the major factors in the absorption of nitrogen oxides by aqueous solutions of nitric acid. From these equations, the coefficient of the absorption rate can be calculated. Use of these also permits calculation with adequate accuracy of the dimensions of a horizontal mechanical absorber for production of nitric acid. 51/ The efficiency of absorption of nitrogen oxides is important in production of nitric acid.

The effectiveness of a revolving absorber for the absorption of nitrogen oxides by sulfuric acid was also investigated by Ganz. The absorption efficiency of the nitrogen oxides increased with the peripheral velocity of the plates, with the acid concentration, and with the nitrogen oxide concentration. It decreased with an increase in the rate of gas flow and the acid temperature. 52/

Solutions of ferrous sulfate have been used to decontaminate gases containing nitrogen oxides and to return the nitrogen oxides (concentrated) into the production process. Ganz and Mamon studied this system of chemisorption. They showed that the coefficient of absorption of nitric oxide increased in proportion to the 0.8 power of the gas velocity, and that the gas film coefficient was controlling. The design of industrial absorption equipment was simplified by application of these data.

53/ They further proved experimentally that vigorous mixing of the nitric oxide and ferrous sulfate solution in mechanical absorbers markedly increased the absorption rate. This improved the efficiency and made the

process of practical importance for the recovery of nitric oxides and for the elimination of air pollution. 54/

The influence of hydrodynamic conditions on the rate of absorption of nitrogen oxides by the milk of lime was studied in a high-speed mechanical absorber in a semiworks unit by Ganz, with the use of industrial gas from a nitric acid plant. He found that the absorption rate increased rapidly with the increase of the peripheral mixing disc speed up to an optimum value. Above this value the rate decreased. The space velocity of the gas was another important hydrodynamic factor that determined the rate of the process. The rate rose rapidly to a maximum with the increase of the space velocity to an optimum value; it then decreased. 55/ These results seem to indicate the practical application of the Soviet surface renewal theory of absorption.

Numerous investigators have shown that the most effective way of accelerating mass transfer processes between gases and liquids in direct contact is to increase the turbulence of the system. Ganz correlated the data of these investigations to derive general laws and design characteristics of high-speed rotary absorbers. He showed that the following hydrodynamic conditions are most favorably obtained by highspeed rotary absorbers: extensive area of phase contact, impact effect of the drops, intensive friction between the gas and the liquid, and rapid hydrodynamic transfer of the interacting components. 56/

A. N. Fernovskaya and A. P. Belopol'skiy found the absorption of sulfur dioxide in water was decreased by surface-active agents. This decrease was independent of gas velocity at constant liquid rates and was not affected by temperature. The surface-action agent modified the hydrodynamic conditions in immobile layer of the liquid for liquid film controlling diffusion. 57/ For absorption of ammonia in water, in which system the gaseous diffusion layer was controlling, they learned that no reduction in the rate of absorption was caused by surface-action agents. The reverse was true for carbon dioxide-water systems, in which the liquid diffusion layer was controlling. 58/ These results indicated the possibility of a method of determining which layer, i.e., gas or liquid, was diffusion controlling in any given system.

A series of studies were carried out by T. A. Sarukhanyan and Belopol'skiy on the absorption of carbon dioxide by aqueous ammonia solutions. Their experimental data showed that the absorbed carbon dioxide was mainly consumed in the reversible reaction of ammonium carbonate formation and that, at any given instant, an equilibrium existed between carbon dioxide and ammonia. 59/

I. N. Kuz'minykh et al. presented data on the recovery of nitrogen oxides by absorption in soda and lime solutions. These data showed that increased basicity of the solutions improved efficiency of absorption. 60/ The reaction of sulfur dioxide and ferric sulfate during an absorption process was studied by Kuz'minykh and T. B. Bomshteyn who sought to develop the kinetics of the reaction and to improve the efficiency of recovery. 61/

Absorption of sulfur dioxide in solutions of calcium and magnesium bisulfite solutions is an important phase of paper mill operation. Data presented by Kuz'minykh and M. D. Babushkina were more complete and usable

than those presented by earlier investigators. 62/ Kuz'minykh and several co-workers have been investigating mass transfer efficiency in plate columns. One of their papers reports coefficients of mass transfer in both the gas and liquid phases on horizontal perforated plates at varying gas velocity were measured for the evaporation of water and desorption of oxygen from water solution. Gas velocities up to 1.8 meters/ second were studied and the gas-phase coefficient was reported as proportional to the 0.85 power of velocity. The liquid phase coefficient reached a maximum value of between 0.5 and 0.6 meter/second (gas velocity) and then dropped to a constant value (about one-half the maximum) above 1 meter/second. 63/

In another paper, Kuz'minykh and two co-workers examined the absorption of nitrogen oxides in a plate column. They showed the efficiency coefficient for each plate decreased as the absorbent became richer and the gas poorer in absorbate. 64/ A. S. Bukhman and K. M. Malin investigated the absorption of nitrogen oxides in sulfuric acid. They found that increasing the linear velocity of gas flow increased the rate of gas absorption up to an optimum value, above which the rate remained constant. 65/ These experimental results, well-known to theoreticians, are of practical importance.

Malin and Bukhman applied the film theory to their data on absorption of nitrogen oxides in sulfuric acid. A simple formula expressed their data:

$$Na = Kg + \Delta Pg$$

where, Na was the amount of nitrogen oxides absorbed per unit time; Kg was the coefficient of the absorption rate; and ΔPg was the partial pressure difference for the nitrogen oxides. 66/ Using a wetted wall tower for this system, they proved that the liquid film resistance was negligible. 67/ They also proved that in a wetted wall tower, where the resistance of the gas film was controlling, the driving force for the absorption was the sum of the partial pressures of the nitrogen oxides in the gaseous phase. 68/

Ferric hydroxide has been long used to absorb hydrogen sulfide from commercial gases -- either to purify the gases or to recover the hydrogen sulfide. While the chemistry of this process is well-known, the kinetics of the absorption have been poorly defined. M. D. Kuznetsov and A. Ye. Sagalovskiy studied this process and showed that the absorption of hydrogen sulfide by ferric hydroxide was a heterogeneous reaction, involving kinetic and diffusional stages, that took place with the formation of a layer of reacted material. The principal resistance to the rate of reaction was brought about by the slowness of the diffusion of hydrogen sulfide through the reacted material. 69/

With Ye. V. Popova, these scientists investigated the absorption of ammonia from coke-oven gas by sulfuric acid in a Venturi-type sprayer

apparatus. They found 99.8 percent of the ammonia could be recovered by using a larger amount of absorbent and by increasing the gas velocity in the Venturi spray heads. 70/

G. D. Sirotkin and V.V. Starostin determined the absorption of nitric oxide by aqueous solutions of ferrous sulfate and ferrous chloride and showed the equilibrium constant, K, was expressible by:

$$K = V / (22.4 - V) P_{\text{NO}}$$

where, V is liters of nitric oxide absorbed by one mol of ferrous iron. 71/ Sirotkin's study of the absorption of nitric oxide by the complex of ammonia-cobalt solutions demonstrated the efficiency of this absorbent. 72/

Disc absorbers were designed by N. I. Timkin for sulfur dioxide and sodium sulfide interaction to produce sodium thiosulfate and to replace absorption columns. In comparison, the disc absorbers were more efficient than the towers, even at lower power consumption. 73/

Hydrogen sulfide is industrially recovered in order to avoid its nuisance in the atmosphere and to recover its value. Equilibrium data were determined and presented for the first time by A. G. Leybush and A. L. Shneyerson for the absorption of hydrogen sulfide from its mixture with carbon dioxide in monoethanolamine and diethanolamine. They showed that hydrogen sulfide could be removed from the gas to a content of less than 0.0002 percent. 74/ This would give a good recovery system. In order to remove and recover hydrogen sulfide, an investigation was made by I. L. Mar'yasin on the effect of various factors on the efficiency of purification of waste gases from gasoline refineries. His results indicated that the temperature of the system and the method of absorption were the most important variables. 75/ M. S. Litvinenko and S. P. Lundin calculated and correlated the free energies of the reactions of various absorbents for hydrogen sulfide with absorption-desorption efficiencies. They concluded that the optimum absorbents would have free energies between +15.6 and -1.0, preferably near zero. 76/ S. A. Bagaturov found gasoline (petrol) is stabilized in a combination absorption-desorption system, wherein light hydrocarbon fractures are removed. 77/ G. V. Ponomarev developed equations for designing such systems of columns in multicomponent absorption and desorption processes. 78/

V. B. Fal'kovskiy, Yu. I. Mel'nikov and A. V. Ventrova studied the effect of the dimensions of a column and of the gas velocity on the following important processes: absorption of ethylene and propylene in the alkylation of benzene; polymerization of isobutylene in sulfuric acid; and absorption of oxygen by aldehydes in the presence of manganese peracetate. Data were presented and correlated. 79/

Utilizing plan-parallel iron plates as packing, Ya. V. Shvartshteyn carried out an experimental study on the recovery of nitrogen oxides from the waste gases of sulfuric acid production by absorption in sulfuric acid. His results showed this packing was about 3.5 times more economical than ceramic ring packing. 80/

Many more references were reviewed. Since they offered no new concepts and did not advance the state of knowledge of absorption, no direct mention was made of them. In summary, the Soviet work is good theoretically and practical in its implications.

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ADSORPTION

Adsorption -- the process by which a solid surface attracts and holds molecules or ions from a gaseous or liquid phase in contact with the solid -- was first discovered and reported late in the 18th century. However, it remained a laboratory curiosity until the early 1900's, when systematic study of adsorption phenomena developed as part of the general growth of physical chemistry. Early pioneers in this area were J. W. McBain in England, H. Freundlich and M. Polanyi in Germany, and Irving Langmuir and Hugh S. Taylor in the United States. Since 1930, significant contributions have been made by Paul H. Emmett, Stephen Brunauer, Edward Teller and Henry Eyring working in the United States and by A. A. Balandin, M. M. Dubinin and A. V. Kiselev in the Soviet Union. Brunauer presents an excellent historical development of theory and early research in adsorption. 1/ In recent years, adsorption has achieved industrial importance in gas and liquid purification and fractionation processes, and as a vital intermediate step in heterogeneous catalysis.

Adsorption is a very popular subject of research in the Soviet Union, and several institutions of higher learning maintain adsorption laboratories. 2/ USSR scientists are not only abreast of the latest developments in this field, but several of their number have achieved international prominence. Their best work is of excellent quality, and they are adept at applying mathematical analysis and the latest experimental techniques to fundamental studies of adsorption. However, many Soviet papers in adsorption are in the category of data collection, with considerable repetition of subject matter and painstaking pursuit of detailed behavior of various adsorbents and adsorption systems. Theoretical papers are often cryptic and many times offer only a minimum of supporting data. Study of gaseous systems is emphasized, with relatively little work on adsorption from liquids. Further, there is little orientation toward the design and development of industrial processes or equipment, so that the Soviet effort in adsorption is more properly described as physical chemistry rather than chemical engineering.

Recent and current Soviet work in adsorption will be discussed under six subject headings: (a) studies of adsorbents, (b) adsorption equilibria and rates, (c) adsorption in catalysis, (d) chemisorption, (e) experimental techniques, and (f) industrial applications.

Studies of Adsorbents

Several Soviet investigators have made systematic studies of adsorbent structure and its relation to adsorptive properties. Typical of this work is the research on silica gels under the general direction of A. V. Kiselev at Moscow State University and the studies of activated carbons by M. M. Dubinin and associates at the Institute of Physical Chemistry, Academy of Sciences, in Moscow. Other Soviet investigators who have contributed significant research in this general area are I. Ye. Neymark at the Institute of Physical Chemistry im. L. V. Pisarzhevskiy, Academy of Sciences Ukrainian SSR, who has studied silica gels and various modified

silicates; S. P. Zhdanov at the Institute of Silicate Chemistry in Leningrad, a specialist on porous glasses; and V. T. Bykov at the Far Eastern Affiliate im. V. L. Komarov, Academy of Sciences, who has investigated several natural adsorbents..

A. V. Kiselev, a very prolific writer, has averaged over 10 publications on adsorption annually since 1950. Most of his papers list co-authors, presumably colleagues and students. He also writes extensively on other aspects of physical chemistry, notably kinetics and catalysis. A leading exponent of the "adsorptive-structural method" of studying adsorbents, Kiselev set forth his basic approach to this problem in a fundamental paper published in 1949. 3/ In this work he classifies adsorbents into four structural types, based on their specific surface, pore volume, and pore size distribution, which he supports with representative isotherm data. Kiselev and his students have centered their attention on silica gels, and they have published numerous articles relating gel structure to adsorption properties. 4/ 11-13/ These papers tend to be somewhat repetitious and to review earlier work, but most of them report at least some new significant data.

As this work progressed, Kiselev recognized the significance of chemical as well as physical properties of adsorbents. 14-18/ His current experimental research deals with the effects of gel hydration on adsorptive properties, particularly toward water, alcohols, and other compounds capable of forming hydrogen bonds. 17/ 18/ 18a/ 19-21/ In addition to silica gel, he is also studying the structure of aluminum silicate catalysts, activated carbons, and magnesium hydroxide. 22-29/ Kiselev works primarily with gas adsorption, but he has also investigated the influence of adsorbent structure on adsorption from solutions by both silica gel and activated carbon. 30-35/ In a recent paper Kiselev described a series of experiments in the modification of adsorbent surface structure by means of chemical reactions. 36/ He found that reaction with alkylchlorosilanes removes surface hydroxyl groups and renders silica gel hydrophobic. Oxidation of activated carbon by treatment with NaClO_2 forms surface carboxylic and phenolic groups which greatly enhance its affinity for water and other polar adsorbates.

Kiselev's early approach to the problem of adsorbent structure was largely experimental and empirical, and he inferred structural configurations from measurements of adsorption-desorption isotherms and heat effects. Later papers report confirming structural data obtained from X-Ray and electron-microscope measurements. Currently he is studying adsorbent structure theoretically as well as experimentally, suggesting methods for calculating and predicting adsorption forces from structural characteristics. 37-40/ This development parallels the general evolution of adsorbent studies in other countries, but Kiselev has been somewhat slow in introducing theoretical justification of experimental results.

M. M. Dubinin is probably the Soviet specialist in this area who is best known abroad. He has published widely on various aspects of adsorption, but his chief work has been a continuing investigation of activated carbons. Some of his studies have been largely experimental, consisting of the collection of extensive equilibrium data with numerous adsorbates and empirical correlation of these data by means of various existing isotherm equations. 41-47/ However, Dubinin recognized the importance of theory even in his early

work, and his more significant papers stress the application of basic fundamentals and mathematics to the elucidation of adsorbent structure and adsorption phenomena. Based on the Polanyi theory of adsorption potential 1/ 48/ Dubinin developed new isotherm equations, suggesting that adsorbent as well as adsorbate properties must be taken into account. 49-52/ He classified active carbon porosity into three types, according to size and adsorption characteristics. 53-55/ In "micropores" (diameter substantially less than 50 microns) he found adsorption to be proportional exponentially to (log pressure), 2/ while in "macropores" (1000 microns and larger) only to log pressure. Adsorption in intermediate pores follows an intermediate relationship. These findings are well summarized in a series of very similar papers published both in the USSR and abroad. 56-60/

Dubinin has also made an extensive study of carbon surface oxides and their effects on adsorption of various vapors. 61-68/ Capacity for adsorbing non-polar gases was found essentially independent of surface oxidation, whereas surface oxides greatly increased the adsorption of water vapor and other polar substances. Dubinin reasoned that oxides serve as active centers capable of forming hydrogen bonds and creating "islands" of a two-dimensional condensed phase which eventually coalesce into a monolayer covering the whole surface. He substantiated this theory with measured heat effects closely approximating heats of liquid condensation. By postulating a definite surface mechanism, Dubinin takes issue with the "American School", which attributes isotherm discontinuities to capillary condensation. He acknowledges, however, that the problem is not completely solved and proposes further study via other chemical treatment of carbon surfaces and microwave spectroscopy of bonds. 68/

Lately Dubinin has returned his attention to the more general problem of carbon porosity. He is studying more rigorous mathematical methods of calculating pore volume distribution, 69-71/ including statistics. 72/ He is reanalyzing data obtained earlier in an attempt to develop more precise isotherm equations and to correlate both adsorbent and adsorbate properties. 73-75/ Recently he introduced the concept of branched porous structures, suggesting a more sophisticated approach than evidenced in some of his earlier papers. 76/ As this work progresses, it should further enhance Dubinin's established international stature as an expert on adsorbent structure.

The work by I. Ye. Neymark on adsorbent structure started with studies of silica gel porosity in relation to benzene and methyl alcohol adsorption characteristics. In 1947 he reported that the pore volume of silica gel was increased by raising the surface tension of the acid coagulant used in precipitating the gel. 77/ He classified gels into three types. (microporous, medium, and coarse) and showed that adsorption hysteresis and capillary condensation increase with pore size. 78/ 79/ He suggested that on microporous gels benzene adsorbs as a vapor, but on coarse gels as a condensed liquid. 80/ Neymark found that silica gel porosity could be affected by the purity of wash water used in its preparation; gels washed with distilled water had finer pores than those washed with tap water containing calcium bicarbonate, which partly transformed the surface to calcium silicate. 81/ 82/ Results such as these led him to experiments in modifying the surface of silica gel with hydrophobic ions 83-85/ and to the preparation

and study of various gel mixtures and complex silicates. 86-91/

By co-precipitating alumina and silica, Neymark has been able to make gels with porosities intermediate between pure silica and alumina gels. 86-87/ A new family of gels was prepared by adding titanium oxides to sodium silicate solutions, followed by precipitation with sulfuric acid. 88-89/ Recently Neymark and co-workers found that the adsorption activity of alumina gels decreased with "ripening" in the mother liquor; this effect is paralleled by transformation from the amorphous to the crystalline state, as confirmed by X-ray analysis. 90/ Neymark's latest work concerns the preparation and characterization of ferrogels of varying porosities 91/ and of silica gels modified with nickel, magnesium and titanium hydroxides, in which he reports that alcohol treatment during preparation and drying increases gel porosity. 92/

For several years, S. P. Zhdanov has been studying the structure of "porous glasses" made by treating crushed glass or quartz with strong alkali solutions. His methods involve adsorption measurements, crystallography, and electron microscopy. 93-95/ 101-102/ Recently Zhdanov has been concerned primarily with surface hydration and its relation to chemical structure. 96-100/ His results and their interpretation are in general agreement with the work of Kiselev, as pointed out in a recent joint paper. 98/ Zhdanov postulates that surface hydroxyl groups are largely responsible for the adsorptive properties of porous glasses 99/ and that every adsorbed water molecule must be "connected with" two hydroxyl groups. 100/ Thus dehydration of a silicate adsorbent affects the nature as well as the extent of its active surface.

S. A. Levina and N. F. Yermolenko at the Institute of Chemistry, Academy of Sciences Belorussian SSR, have been studying the structure and adsorptive properties of various gels in relation to precipitation conditions and drying methods used in their preparation. They are concerned primarily with aluminum, chromium and iron hydroxides, 103-106/ although a paper on cadmium hydroxide has also appeared. 107/ In collaboration with Z. A. Krivchik, Yermolenko has also investigated the steam activation and heat treatment of various commercial charcoal adsorbents. 108/

V. A. Kargin at Moscow State University, a leading Soviet specialist in polymer research, has used adsorption as a means of identifying polymer structure. For example, he has distinguished between dimer and crystalline polyamide by means of formic acid adsorption. 109/ From data on adsorption of hydrogenated monomers on a variety of amorphous vinyl and acrylic resins, Kargin has studied changes in molecular packing density in vitreous-elastic transitions. 110/ He has compared synthetic and natural rubber structures by means of hexane adsorption. 111/ A summary of Kargin's findings concerning polymer structure and adsorption characteristics was published abroad in 1957. 112/

A number of Soviet adsorption specialists have been cataloging the properties and structure of natural adsorbents native to their country. Typical of this effort is the work of V. T. Bykov, who has studied systematically various diatomites, clays and weathered earths from different areas of the Soviet Union. 113-116/ Adapting some of Dubinin's

techniques and isotherm equations, Bykov has studied porosity by capillary condensation and mercury pressure methods 117-118/ and developed a dynamic procedure for measuring adsorption curves, whereby a standardized organic solution is passed through a column of adsorbent and the effluent is analyzed in small increments. 119-120/ Other recent Soviet investigations of natural adsorbents include studies of several Georgian clays of the bentonite series by G. V. Tsitsishvili; 121/ of dark green Odessa earths by S. I. Burshteyn and associates; 122/ of Chenushy bentonite clay by V. Bregvadze; 123/ of Volga diatomites by F. A. Slisarenko and co-workers; 124/ and of several kaoline and montmorillonite clays by M. I. Kuadzhe. 125/

Adsorption Equilibria and Rates

Several Soviet investigators have contributed significant information to the literature on adsorption equilibria and rates, often as a by-product of other work. For example, M. M. Dubinin's continuing research on activated carbons has brought him international recognition as a leading exponent of the potential theory of adsorption and an expert on adsorption isotherms of various materials on carbon surfaces. 46-47/ 49/ 50-52/ 57/ 61/ 65/ 66/ 71/ 75/ Similarly, A. V. Kiselev has made extensive use of isotherm measurements in his studies of adsorbent structure and consequently published data on adsorption equilibria of nitrogen, water vapor and various organic materials. 5/ 9/ 11-13/ 17/ 19-21/ 23-25/ 28/ 32-34/ Kiselev has also participated in detailed analysis of low-temperature nitrogen isotherms on various adsorbents, 126/ 127/ and in the development of an ultrasensitive automatic calorimeter for measuring heats of adsorption. 128/ With the aid of this device, Kiselev and A. A. Isirikyan have been making a continuing investigation of heat effects in the adsorption of various hydrocarbons. 129-133/ Based on the heat effects involved, they picture physical adsorption as a three-step process; first a reversible adsorptive process until the pressure is reached where desorption hysteresis begins, then a region of capillary condensation with hysteresis, and finally an area of reversible capillary condensation. 129/ They also suggest that heat of adsorption for normal alkanes varies linearly with the number of carbon atoms per molecule. 133/

N. N. Avgul' has appeared as the principal author in a series of significant joint papers with Kiselev and other colleagues at Moscow State University dealing with the adsorption thermochemistry. By measuring differential heats of adsorption of hydrocarbons, water, and alcohols on carbon black, Avgul', O. M. Dzhigit and Kiselev conclude that on porous carbons, hysteresis and capillary condensation are involved, whereas on non-porous carbon, unimolecular adsorption via hydrogen bonding predominates. 134-138/ On silica gel, they found that the capillary condensation theory and differential heat measurements enable simple calculation of surface areas that agree closely with more conventional area determination methods. 139/

In collaboration with several others, Avgul' and Kiselev have extended their calorimetric studies of adsorption to support more general considerations of thermodynamics and adsorption forces on active carbon surfaces. Experimental data are reported in close agreement with calculated

values of free energy and entropy of adsorption for benzene, 140/ nitrogen, and ammonia, 141/ a series of normal and branched alkenes, 14-144/ and various non-polar molecules. 145/ Influence of chemical structure and porosity of the carbon surface was also studied. 146/ 147/ The most recent paper by these authors concludes from entropy curves that isoalkanes and naphthenes are adsorbed on carbon black in a dense layer closer to the liquid state than adsorbed normal alkanes; an additive scheme is proposed for calculating heat and free energy of adsorption of branched hydrocarbons. 148/ This work appears to be of excellent quality and demonstrates that Kiselev, Avgul' and their colleagues at Moscow State University are well versed in both the theory and practice of modern adsorption thermodynamics.

Two other highly competent Soviet specialists in this area are B. P. Bering and V. V. Serpinskiy of Dubinin's staff at the Academy of Sciences' Physical Chemistry Institute in Moscow. Several years ago they developed an equation of state for adsorbed molecules in a monomolecular layer. 149/ More recently they have derived simple equations for heat and entropy of adsorption, respectively:

$$\begin{aligned} \bar{E} &= \alpha RT + a \left(\frac{\partial \ln h}{\partial \ln a} \right)_T - RT \ln h \\ \Delta S &= \frac{\partial \bar{E}}{\partial T} = -\alpha RT \left(\frac{\partial \ln a}{\partial \ln h} \right)_T \end{aligned}$$

where \bar{E} is the Polanyi adsorption potential ($= -RT \ln h$), a is the quantity adsorbed, α is the volumetric coefficient of expansion of adsorbate and h is the pressure ratio ($= P/p_{sat}$). These equations are claimed to agree closely with calorimetric data from the literature and to permit calculation of differential heats of adsorption from a single isotherm curve. 150/ 151/

Bering and Serpinskiy have also collaborated on some interesting experimental studies. They developed a quartz microbalance of high sensitivity, which enable precise isotherm measurements at low pressures. 152/ 153/ They investigated carbon dioxide adsorption on silica gel at pressures up to 85 atmospheres and found evidence of a liquid-state adsorbate at temperatures below the critical. 154/ They also published a series of papers on the adsorption of gas mixtures, but appear to have concluded this experimental study without offering any confirmed general theory of such systems. 155-160/ In collaboration with K. A. Ioyleva, Bering published three interesting experimental papers on the adsorption of water and various organic vapors on mercury surfaces. 161-163/ They suggest that isotherm discontinuities result from two-dimensional phase transitions (surface condensation) and report that differential heat of adsorption attains a maximum close to complete coverage of the surface.

Typical of a number of recent Soviet experimental studies reporting gas adsorption isotherms for various systems, are papers on the adsorption of various organic vapors on mica, 164/ and of hydrocarbons on charcoal 165/ and on commercial aluminum oxide. 166/ M. G. Kaganer has studied nitrogen adsorption isotherms at very low pressures down to the region where Henry's law applies. 167/ B. V. Il'in has proposed some semi-quantitative

titative rules concerning molecular attraction in adsorption from data on the heat of wetting of various adsorbent surfaces. 168/ L. V. Radushkevich is seeking to explain capillary condensation phenomena with the aid of calculations on various proposed physical models. 169-172/

Several Soviet workers have been studying adsorption kinetics. Among the principal efforts are those of D. P. Timofeyev and Ya. M. Bikson at the Institute of Physical Chemistry, Academy of Sciences, in Moscow. Timofeyev has studied both adsorption and desorption rates of benzene vapor in thin layers of activated carbon. He has found that adsorption kinetics may be controlled by either external or internal diffusion. 173, 174/ However, desorption rates are generally much slower and controlled by pore diffusion. 175/ By means of X-ray techniques, he is seeking to distinguish between diffusion in the volume of the pores and on their surface. 176/ Timofeyev has suggested a dimensionless ratio dv/D (where d = particle diameter, v = velocity of air-vapor mixture, D = internal diffusion coefficient) as a criterion for predicting diffusion mechanism in adsorption from an air-vapor mixture flowing through a thin bed of adsorbent. He states that external diffusion controls when $dv/D < 5$ and internal diffusion controls when $dv/D > 20$. According to Timofeyev, for values between 5 and 15, the adsorption front inside a spherical particle progresses at approximately constant rate almost to complete saturation. 177/ Bikson has treated mathematically the problem of adsorption dynamics for a binary gas mixture in an adsorbent column, suggesting equations for calculating the length of the working layer under conditions of parallel transfer. 178, 179/ It seems strange that this basically sound work has not been followed up with subsequent papers.

M. V. Tovbin and various co-authors have studied desorption rates from activated carbon into solutions in relation to porosity, grain size and elution rate. Some of this work involves iodine and uses radioactive tracer methods. 180-182/ Tovbin has suggested a self-adsorption mechanism to account for enhanced retention of adsorbate at concave surfaces in capillaries. 183, 184/ He is currently studying the desorption kinetics of acetic acid and other polar organic compounds. 185/

Other recent Soviet studies in this area worthy of note are the work of N. D. Gorchakov and I. I. Pogodin on the rates of stripping various organic solvents from charcoal columns with carbon dioxide gas; 186/ and research on internal diffusion coefficients in porous catalysts by K. N. Bolonogov and B. I. Popov. 187/ K. A. Gol'bert has suggested a method for calculating sorption dynamics for the case where the limiting step is internal diffusion. 188/ Later he applied this approach to the analysis of data on the adsorption of ethylene from binary mixtures with hydrogen, methane and nitrogen. 189/

Interest in developing calculation methods applicable to commercial process design seems to be rather limited in the Soviet literature. P. Ye. Romankov has suggested equations applicable to adsorption and desorption of gases in fluidized beds of adsorbent. 190, 191/ A. N. Planovskiy and L. A. Vlasenkov studies the separation of methane-hydrogen mixtures in a five-stage fluidized char apparatus. They reported that at high saturation levels (0.9 and above) adsorption is controlled by internal diffusion and

at lower levels (below 0.8) by surface diffusion. 192/ N. I. Smirnov and co-authors have derived differential equations describing steady-state adsorption in packed towers, 193, 194/ and extended these to a simplified method for correlating mass transfer coefficients with gas concentration, gas velocity and height of sorbent layer. 195/ Ya. V. Shevelev has offered a general solution for dynamic adsorption in packed beds, but restricted to linear isotherms. 196/ Ye. V. Vagin and A. A. Zhukovitskiy recently published a mathematical paper working out distribution equations for thermo-adsorption separation processes, but again on the basis of linear isotherm behavior. 197/

Although Soviet research in adsorption is concerned largely with gaseous systems, some interest in adsorption from liquids was also noted. Several of these papers appear aimed at processes for recovering and separating metals. For example, I. V. Gebler and K. K. Stramkovskaya reported that copper, silver, and gold are adsorbed from their aqueous solutions by granulated brown coal. 198/ I. Ye. Starik and I. A. Skul'skiy have studied the adsorption of radioactive elements from dilute nitrate solutions on fluoroplast and paraffin surfaces. 199/ S. A. Voznesenskiy and colleagues report that the addition of ethyl alcohol or acetone markedly increases the adsorption of microquantities of Cs^{137} and Sr^{89} from water solution on ultramarine, iron oxide, barium sulfate, and titanium dioxide adsorbents. 200/ I. A. Sheka and B. A. Voytovich have devised a method for separating zirconium and hafnium by silica-gel adsorption from solutions of their tetrachlorides in methyl alcohol. 201/ M. S. Belen'skiy and T. G. Alkhazov investigated the kinetics of silver salt adsorption by activated carbon. 202/ D. N. Strazhesko and collaborators have found that the adsorption of strong acids and electrolytes by carbon from non-aqueous solutions is electrochemical in nature. 203-207/

N. A. Izmaylov has reviewed the theory and practice of adsorptional separations. 208/ I. P. Kutanov has correlated the effects of functional groups on the adsorption of various organic acids from alcohol and acetone solutions on activated carbon. 209-211/ A. N. Kharin has studied kinetics of iodine, 212/ essential oil 213/ and acetic and butyric acid adsorption from aqueous solutions on activated charcoal. 214/ Analytical separations of aliphatic alcohols by means of silica gel 215/ and of normal hydrocarbons via adsorption on carbamide 216/ have been suggested. I. V. Smirnova and K. V. Topchiyeva are studying the adsorption and molecular orientation of various hydrocarbons on alumina and other catalyst materials. 217, 218/

The role of adsorption phenomena in electrochemical kinetics has been reviewed by A. N. Frumkin. 219/ Some recent Soviet papers in this area include a theoretical and qualitative experimental study of adsorption layers on falling drops of electrolyte by S. S. Dukhin and B. V. Deryagin; 220/ investigation of cation adsorption at platinum electrodes by A. D. Obruchyeva; 221, 222/ and studies of anion adsorption by N. A. Balashova 223/ and S. A. Nikolayeva and S. Rakhnu. 224/ B. S. Krasnikov and M. V. Pevnitskaya discuss recent Soviet research on the adsorption of surface-active agents at platinum and copper electrodes. 225/ V. L. Kheyfets and colleagues at the A. A. Zhdanov State University in Leningrad have published a series of papers on experimental studies of the effects of various adsorbed materials on double-layer phenomena at solid metal and

mercury electrodes. 226-230/

Adsorption in Catalysis

Detailed analysis of Soviet research on catalysis is beyond the intended scope of this report. However, several Soviet catalysis specialists have studied the role of adsorption phenomena in catalytic mechanisms, and brief discussion of some of their recent work is appropriate here.

Probably the best-known Soviet expert in this area is A. A. Balandin of Moscow State University. Based upon extensive theoretical and experimental evaluations of activation energies and adsorption forces, Balandin formulated his multiplet theory of surface layers, postulating superimposition of an adsorbed molecule on the lattice of the catalyst. 231-235/ He then suggested rules for relating activation energy effects and layer changes and explaining catalyst selectivity on the basis of chemical properties and adsorption bond energies. 236-238/ Balandin's theories are well summarized in a recent review of his own work. 239/ To facilitate their practical application, he has developed a method for precise determination of relative adsorption coefficients and used it to measure the free energy of adsorptive displacement of butylene by water on catalyst surfaces. 240,241/ He has interpreted kinetic data on catalytic hydrogenation, dehydrogenation and dehydration reactions in terms of adsorption and surface orientation effects. 242-244/ A recent study by Balandin and colleagues of the relative adsorption of various saturated alcohols on oxidizing catalysts concludes that both plane-orientation and normal-orientation exist and suggests a new term "specific effective place" to describe a surface site capable of accommodating a particular molecule. 245/

S. Z. Roginskiy published a book in 1949 on adsorption and catalysis on heterogeneous surfaces. 246/ He has suggested that the catalytic properties of heterogeneous surfaces can be predicted from data on heats of oxygen and hydrogen adsorption. 247/ Later Roginskiy and co-workers showed that metal oxide catalysts which strongly adsorb phenol, promote alcohol dehydrogenation, whereas those which adsorb pyridine readily promote dehydration. 248/ Another paper suggests use of molecular adsorption to evaluate the effect of catalyst additive. 249/ Recently, Roginskiy has been using exchange reactions of adsorbed isotopes to study intermediate compounds on catalyst surfaces. 250/ A colleague of his at the Moscow Institute of Physical Chemistry, Academy of Sciences, N. V. Kavtaradze, is studying hydrogen adsorption heat effects as a means of predicting catalytic activity of nickel, platinum, iron and chromium toward hydrogenation reactions. 251-252/

Another advocate of adsorption as a tool in catalysis research is A. M. Rubinshteyn of the Institute of Organic Chemistry imeni N. D. Zelinskiy, Academy of Sciences, USSR. He has perfected a dynamic method for measuring catalyst areas by benzene adsorption and uses adsorption measurements to study and correlate the activity of various types of catalysts. 253-256/ A. I. Shlygin of Moscow State University has studied gas adsorption effects on the catalytic activity of platinum electrodes. 257-261/

Studies such as these, while not greatly different from contemporary research outside of the Soviet Union, are significant because they show that Soviet physical chemists are capable of applying their extensive knowledge of adsorption phenomena to support their research on catalysis.

Chemisorption

The leading Soviet specialist in chemisorption is F. F. Vol'kenshteyn at the Moscow Institute of Physical Chemistry, Academy of Sciences. He has made several significant contributions to knowledge in this area, starting with his fundamental theory of activated adsorption set forth in a series of papers published in 1952-53. 262-266/ His basic assumption is that free electrons in the adsorbent lattice act as adsorption centers; he shows that the number of these centers per unit surface increases with increasing temperature and increasing coverage. This leads to a mathematical expression for adsorption equilibria approximating the Freundlich isotherm, provided that interaction between adsorbed molecules and their dissociation are absent. Vol'kenshteyn postulated that surface impurities, as well as light, can influence the concentration of adsorption centers, and he conceived of both "donor" and "acceptor" impurities acting in opposite directions. Later he suggested that both "weak" (one-electron) and "strong" (two-electron) bonds may exist; "strengthening" is accomplished by the capture of a free lattice electron by the adsorbed atom and may absorb or evolve heat, depending upon whether or not a "hole" is created in the adsorbent lattice. 267/ Subsequent papers refined this theory to allow for surface inhomogeneity and dissociation of diatomic molecules. 268, 269/ It was extended to provide a theory for the action of semi-conductor catalysts by assuming that only atoms or radicals adsorbed in the "weak" state and displaying unsaturated valences possess enhanced reactivity. 270-273/ Recent experimental data on benzene oxidation appear to confirm Vol'Kenshteyn's theories quite well. 274/

V. L. Bonch-Bruyevich and V. B. Glasko are seeking to develop generalized equations for chemisorption on various types of metals, with the aid of a "Strela" computer. 275/ Recently Sh. M. Kogan proposed a statistical method to calculate the distribution of "weak", "strong" and "strong donor" bonds, to further extend the Vol'kenshteyn theory. 276/

For several years, V. I. Lyashenko and co-workers at the Kiev Institute for Physics, Ukrainian Academy of Sciences, have been studying chemisorption experimentally. Early experiments showed that the adsorption of polar molecules altered the conductivity and work function of copper, zinc and cadmium oxide semiconductors, whereas non-polar molecules had negligible effect. 277/ Similar effects were later observed on molybdenum sulfide. 278/ Adsorbed molecules were also found to alter the photoconductivity of various materials. 279-281/ In a subsequent study, Lyashenko found that the work functions of CuO, NiO, MnO₂ and germanium exhibited maximum values when acting as catalysts for CO oxidation or N₂O decomposition. 282/ Lyashenko and V. G. Litovchenko are currently studying the influence of various polar and non-polar adsorbates on the work function and conductivity of germanium, as well as on the rates of the changes involved. 283/ 284/

Another interesting line of study is suggested in a recent paper by

A. Terenin and Yu. Solonitzin. 285/ They have investigated the action of light on chemisorption and report highly specific effects. Oxygen is strongly phosorbed by silica gel and by ZnO in presence of excess oxygen, but it is photodesorbed from ZnO with zinc in excess. Earlier P. Ye. Val'nov reported that CO is photodesorbed from nickel and H₂O from cadium and zinc, but not from bismuth or antimony. 286/

Experimental Techniques

Soviet researchers in adsorption appear to be thoroughly conversant with the latest experimental techniques in their field.. Their papers frequently mention methods and equipment described in the Western literature and often suggest improvements and refinements. They are adept at using modern instruments, such as the electron microscope, x-ray devices and spectrophotometers, to corroborate evidence inferred from adsorption measurements as to adsorbent and adsorbate properties. Recent literature indicates increasing use of radioactive tracer materials. In short, Soviet scientists appear to be competent and resourceful experimentalists. These conclusions are confirmed readily by the descriptions of apparatus and methods in the experimental studies cited above. In addition, a few more examples will be mentioned.

A. I. Sarakhov has developed a micro-torsion balance with a sensitivity of approximately 5×10^{-8} gram. 287/ Using this device he has been able to study adsorption on relatively non-porous materials with specific surface less than 1 squaremeter per gram. 288/ 289/

Sarakhov has collaborated with M. M. Dubinin in developing a simple low-pressure device for measuing adsorbent porosity by means of mercury. 290/ Rubinshteyn has perfected simplified dynamic methods for measuring catalyst surface areas with physically adsorbed benzene vapor 253/ 291/ 292/ or chemisorbed iodine from CCl_4 solution. 293/ He has also designed a microtome for preparing catalyst sections down to 0.01 micron in thickness for electron-microscope examination. 294/ M. G. Kaganer advocates use of very low pressures in area measurements by the BET method, 295/ and D. P. Dobychin suggests a scheme whereby only one point is necessary in using the BET equation. 296/ B. N. Vasil'yev has designed an adsorption apparatus capable of attaining and operating at 80 atmospheres pressure without using a compressor. 297/

A. N. Frumkin discusses the application of electrochemical techniques in studying surface phenomena. 298/ L. N. Kurbatov reviews the dielectric properties of adsorbates on silica gels. 299/

S. Yu. Yelovich and L. Ya. Margolis describe an experimental method for measuring surface conductivity of adsorbents. 300/ I. V. Zhilenkov suggests using the Debye dispersion effect to study water adsorption on silica gel. 301/

Several analytical methods based on adsorption are mentioned in recent Soviet literature. Development of gas chromatography in the Soviet Union is credited largely to A. A. Zhukhovitskiy, presently occupying the chair of Physical Chemistry at the Moscow Steel Institute. 302/ He has

worked out the theory of thermo-adsorption separations 303/ and recently patented a laboratory apparatus for continuous separation of gases. 304/ A. M. Brodskiy and colleagues have described an apparatus capable of semi-quantitative separation of a 12-component mixture 305/ and described its testing with radioactive tracers. 306/ P. N. Fedoseyev has suggested alkaline-earth silicates as adsorbents for SO₂ in combustion analysis of organic substances. 307/

Industrial Applications

The application of adsorption techniques to large scale fractionation of gases and liquids is poorly developed in the Soviet Union. Recent literature shows increasing Soviet interest in this area, but the USSR appears to be considerably less advanced than the Western world in both processes and equipment.

Use of activated carbon for the purification of gases has been practiced in various Soviet industries for a number of years. 308/ Improvements in oxygen production have been achieved by adsorptive drying of air with alumina, 309-311/ and by removing acetylene and carbon dioxide via low temperature adsorption on silica gel. 312/ 313/ Recently a new process was patented for recovering and concentrating krypton and xenon in oxygen plants through silica gel adsorption. 314/

Continuous moving-bed adsorption processes are relatively new to Soviet industry. As recently as 1958, there were none in operation, 315/ but there are at least some process developments in the laboratory stage. N. V. Kel'tsev has described an experimental moving-bed apparatus for drying gases 316/ and another for separating acetylene-carbon dioxide mixtures. 317/ Kel'tsev and two colleagues have patented a process for continuous adsorptive fractionation of butane-butylene mixtures with silica gel. 318/ A recent article by P. Benedek and co-workers describes an experimental hypersorber for separating mixtures of hydrogen, CO, CO₂, and acetylene, but admits that it copies the Berg process (American). 319/ Some originality is shown by L. A. Kul'skiy and colleagues in their design of two-stage countercurrent adsorber for recovering phenol from water solution. 320/

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CRYSTALLIZATION

The importance of crystallization studies and the over-all objectives of the Soviet scientists in this area were succinctly stated by Academician A. V. Shubnikov at the First Convention on Crystal Growth held in Moscow on 5-10 March 1956. 1/

Crystal formation problems are of great interest (importance) in science and technology. Modern solidstate physics (piezo- and ferroelectricity, ferromagnetism, luminescence, photoelectricity, semiconduction, optics of new crystalline media) is closely bound up with the study and use of large uniform crystals, the growing of which demands a many-sided and profound study of crystallization processes. Crystal studies are also very important in biology, in particular in virology. In contemporary metallurgy and in the chemical, ceramic, etc. industries, improvements to a large extent depend on a profound study of crystallization processes.

N. N. Sheftal, at the same conference, said:

The industrial uses of synthetic single crystals have greatly increased in the past 30 years. This has stimulated much theoretical and experimental work on crystal growth. The theory of the ideal perfect, and later that of the ideal imperfect growth of crystals answered many of the most important problems of growth and dissolution mechanisms in crystals. New powerful methods of studying crystal surfaces were evolved, such as the electron microscope, field-emission microscope, and various interferometers. Fluorite and quartz in no way inferior to the natural have been grown in the laboratory and many new single crystals of artificial compounds have been grown. Finally, natural diamonds have been successfully synthesized.

In spite of these successes we still lack a theory of real crystals, and there is a large divergence between theoretical and practical work on the growth of crystals. 2/

The Soviets engaging in crystallization have made extensive use of Western work as a basis for more rapid advancement of their own studies. Their familiarity with Western scientific publications is shown by numerous references to the outstanding theories and publications of Western scientists in the field. In a typical gesture, the Soviet authors insist upon asserting that their Soviet researchers have further developed and extended any work started in the Western world.

In 1941, V. D. Kuznetsov reviewed the completed and continuing work on the physics of solids in the various research institutes of the Soviet Union. The main topics of study were the crystallization of supercooled (crystallizing) liquids, crystal growth and habit, plasticity and strength of ionic crystals, plastic deformation of polycrystalline solids (metals), brittleness of steels and alloys, physical foundation of metal cutting, and metallic and nonmetallic mono- and polycrystals. With the exception of an increased emphasis on semiconductor crystals, current studies have been continued on the above or similar topics. 3/

According to A. Novikov, "The Institute of Crystallography, Academy of Sciences, USSR, is the only institution in the USSR which investigates in detail the structure and physical properties of crystals, does research on phenomena taking place in them, and conducts work pertaining to the industrial application of results obtained in this field." Z. Pinsker is director of the Laboratory of Electronography, which is concerned with investigating semiconductor materials and heat resistant alloys, defining the (surface) structure of crystals, and elucidating the forces of interaction between atoms in crystals. At this Institute, studies on crystals for scintillation counters are emphasized. These counters are used in prospecting for nuclear ores and in core sampling in test petroleum wells. Work on crystallized corundum, for instance, rubies, has shown sufficient promise to commence the production of artificial rubies for watches and other instruments, i.e. jeweled bearings. Quartz for spectrographic prisms and lenses has been artificially grown under the direction of N. N. Sheftal. 4/

It is apparent that the Soviet research scientists are encouraged to publish results of their studies that are considered pertinent to continued progress. Some of the publications, however, are essentially repetitious. While this indicates a continuing interest in the field, it also shows the pressure for publishing. For example, Yu. M. Zhvirblyanskiy divulged a method for the crystallization of massecuite from the second syrup of sugar refining (impure molasses). 5/ In 1949, he and A. K. Volobuyeva and D. R. Abragam discussed the kinetics of sucrose crystallization from impure sugar solutions. 6/ In several later reports (1953 and 1955), the same authors presented the information on the same general topic, namely the possibility of obtaining crystalline sucrose by cooling the impure syrup and seeding it with highly dispersed crystallites. 7/

Although many of the Soviet studies are based on Western work and the pressure to publish causes repetition, it should be stated that the Soviets are devoting themselves to intensive studies in this area. To provide a more detailed view of Soviet advances in the crystallization field, the following discussion is organized under four subject headings: crystal growth, crystal systems, habit modification of crystals, and crystallization apparatus and methods for growing crystals.

Crystal Growth

There are presently two main theories of ideal crystal growth. The first is the Kossel-Stranski molecular-kinetic theory of ideal perfect growth; the second is the theory of ideal imperfect dislocation growth. Essentially, the two theories supplement each other, although the rapidly developing dislocation theory relies somewhat on the Kossel-Stranski one for its complex physico-mathematical and structural geometry apparatus. The first theory ignores crystal imperfection and introduces a number of simplifying assumptions in analyzing growth processes. The crystal surface is considered undeformed, and the growth is assumed to occur at very low supersaturation.

The second theory incorporates initial imperfections mainly as screw dislocations. The steps, which gradually rise from the crystal faces, being produced by dislocations, remove the need for two-dimensional nuclei and cause the low-index-faces to grow continuously in a spiral fashion.

While this theory implies that the supersaturation at which growth starts in a perfect crystal should be 50 per cent sufficient dislocations present on the faces of a crystal permit growth at 1 per cent supersaturation.

Sheftal' has stated that the history of an individual crystal can be described in terms of normal development in favorable conditions and complex abnormal development as influenced by unfavorable conditions due to the action of the medium. From initiation via growth and destruction of a single uniform crystal, one passes to skeletal and finally to dendritic growth. At this point, the medium is incorporated to such an extent that the crystal is not completed. 2/

The manner in which the physical properties of crystal depend on the growth conditions is determined, in essence, by the same factors that control the shape. The properties depend on the impurities, i.e. on inclusions of the medium, on the stresses caused by unevenly distributed medium inclusions, and on the supersaturation at which growth occurs. Choosing conditions for growing crystals of a substance with pre-set physical properties amounts to producing a favorable chemical environment, eliminating harmful impurities, using the best type of stirring, providing uniform material supply to the surfaces, preventing impurities from being incorporated, and using carefully controlled temperature gradients. 2/

D. S. Kamenetskaya has conducted studies on the effect of impurities on the production of crystallization nuclei in supercooled liquids. 8/ Her work was based on Volmer's theory of crystallization. This in turn was derived from Gibb's thermodynamic work as extended by Frenkel. 9/ V. I. Danilov's work, as that of other Soviet and foreign workers, has confirmed this theory. Kamenetskaya's results may be summarized as follows: (1) Crystallization centers form impure liquids at high degrees of supercooling that are determined by the surface tension at the crystal liquid interface. (2) The absence of much supercooling in practice is due to active insoluble particles and small amounts of surface-active impurities. (3) The mechanisms that determine the activity of insoluble and soluble impurities differ: (a) the insoluble impurities act as pre-existing centers, (b) small amounts of soluble impurities materially influence the rate of center formation, which is a consequence of their influence on the surface tension and on the activation energy of center formation, and (c) surface-active additives can conceal the actions of insoluble active particles as the liquid may then crystallize spontaneously (throughout the volume) at small degrees of supercooling. 10-14/

Research by K. M. Gorbunova indicates that crystal growth during electrolysis follows laws very similar to those obeyed by metal crystal growth from vapors or by salts from solution. This is true with regard to both the formation of the first nuclei of the new (solid phase) and to the growth of the whole crystal or any of its individual faces. Theory and experiment show that diffusion factors play a large part in the crystal growth mechanism. In the steady state in electrocrystallization, Gorbunov showed that where a single crystal (or a system of crystals not in contact) is growing, the current density (per unit of growing surface) remains constant. 15/ Gorbunova, O. S. Popova, A. A. Sutyagina, and Yu. M. Polukarov have demonstrated that regularities in the growth of cathodic metal deposits, either as dense coatings or friable dendritic structures, can be obtained by varying the deposition conditions. Technically useful properties of such crystalline metals such as high hardness, wear resistance, and defined electrical and magnetic properties can be realized. 16/

The terms "physically possible" and "physically impossible" were first used in relation to the crystal faces by A. V. Shubnikov. By the term, "physically impossible," he referred to the faces of a substance which do not appear, even temporarily, on crystallizing spheres. 17/ O. M. Ansheles also discussed this subject and stated ". . . those faces are physically possible (under given conditions) in which the interparticle distances are equal to or less than the greatest distance of effective interaction between crystals and liquid- or gas-phase particles in at least two directions. Faces not corresponding to this condition are physically impossible." 18/

A. I. Landau has derived equations in partial derivatives that describe the impurity distribution in the melt at any time. The distribution of the impurity in the melt and consequently in the crystal depends primarily on the rate of impurity diffusion in the liquid. Any method of controlling the actual distribution of impurity between the melt and the crystal is highly important in both the growth of semiconduction crystals in their subsequent commercial use. 19/

Electron and ion field-emission microscopes (electronic microscope-projector) have become widely used in the USSR and in other areas of the world for physics research. A. P. Komar and Yu. N. Talanin have used this technique for studying the effect of two-dimensional crystals transformed to liquids on heating (contaminants) in contact with other crystal surfaces. 20/ The device is also valuable for studying certain properties of crystals such as surface migration, adsorption, crystallization, recrystallization and phase changes.

Mechanical crystallizers, i.e. units in which crystallization is aided by stirring, are now commonly used in large scale chemical industry. Although these crystallizers are widely used, their design is based on extremely simplified assumptions since moving or stirred media have not been extensively studied. L. N. Matusevich studied the crystallization of potassium nitrate and potassium ferrocyanide from water solutions in cooled and stirred vessels. He showed that the mean crystal size decreases smoothly as the stirring speed is increased, and that the degree of supersaturation required for crystallization also falls with an increased stirring speed. 21/

Screw dislocation in the growth of B-methyl-naphthalene from alcoholic solutions and from the melt was studied by M. I. Kozlovskiy. His data show that screw dislocations may be formed as a result of the inclusion of foreign particles during crystal growth. 22/ In a further study, he observed that spiral growth occurred most frequently from pure solutions at the edges of the crystal regardless of the rate of growth. 23/

Matusevich also studied the effect of rates of cooling and stirring on the unseeded crystallization of saturated solutions of potassium nitrate and ferrocyanide. His results showed that the temperatures of initial crystallization were not a function of the rate of cooling if high rates of stirring were maintained. When this is applied to commercial crystallization, it means that more rapid cooling results in more rapid crystallization only if more rapid stirring is concomitant. 24/

The kinetics of crystallization of supercooled organic liquids, viz., piperine, salol, and p-toluidine, were defined by G. L. Mikhnevich and V. P. Yefimova. The number of centers depends on the character of the saturation curves and on the rate of cooling. The presence of particles of active admixtures improves the rate of crystallization. 25/ 26/ Under the usual purification conditions, slowly crystallizing organic compounds such as betol, piperine, and aspirin were found to crystallize out on particles of impurities. They contain a crystalline layer of the compound on their surfaces. The kinetics of growth are those of a first order reaction. 27/

The rates of crystallization of potassium aluminum sulfate, copper sulfate, and strontium sulfate were determined by N. A. Figurovskiy and T. A. Komarova. For the first two (highly soluble salts), the rate of crystallization is a second order process which is expressed by the equation

$$\Delta Pi / S \Delta t = kX^2$$

where, ΔPi is the increase in crystal weight, S is the crystal surface, Δt is the time interval, X is the degree of supersaturation and k is a constant. Crystallization does not begin spontaneously for strontium sulfate (slightly soluble salt) until a high degree of supersaturation has been achieved. 28/ 29/

N. V. Alyavdin, Sheftal', and Z. I. Frolova have proved that high growth rates for Seignette salt (double tartrate of sodium and potassium) crystals can be obtained by high supersaturation and elevated temperatures. By destroying the tartrate seeds, which cause decomposition of the Seignette salt at elevated temperatures, single crystals weighing almost one kilogram were grown within 63 hours as compared with the old rate of 12 to 13 days. 30/

Five types of crystallization of isomorphic salts from aqueous solutions have been described by S. M. Chirkov.

- (1) The crystallization path proceeds from the less soluble salt to the more soluble salt independently of the initial composition of the solution. The salts which are finally crystallized are pure.
- (2) The direction of crystallization depends on the initial composition. As the crystallization process proceeds, the solution approaches that of a pure solution of one compound.
- (3) The direction of crystallization depends on the initial composition. As crystallization proceeds, the solution composition approaches that of maximum combined solubility.
- (4) The crystallization path is similar to (1) but the composition of the solid phase varies with that of the mother liquor.
- (5) The crystallization path is similar to (3) but the end composition is different for the two directions of approach. 31/

The factors affecting the crystallization process, i.e. crystal growth, were determined by Sheftal' to be the degree of association, the presence of admixtures, the crystallization pressure, the conditions of equilibrium, and the forms and the solubilities of the crystals.

In 1938, Sheftal' developed a method for growing large sucrose crystals that are heavier than 200 grams by slow cooling over a period of several months. In this way, a very low degree of supersaturation was maintained. He applied his method to the production of large crystals (1.4 kilogram) of Rochelle salt. This salt was badly needed but was not being produced in the Soviet Union prior to 1939. By 1946, however, 75 per cent of the production of Rochelle salt required by the USSR was produced by Sheftal's static method. 33/ During his study on the production of large single crystals, he found that inhomogeneities are caused by foreign inclusions, temperate changes, and uneven growth processes. 34/ The study of techniques for growing large crystals was continued by S. K. Popov and Sheftal' who developed a process for growing monocrystals in a seeded crystallizer for use in optics and piezoelectrics, and for other purposes. The crystallizer is imparted a random motion in relation to the crystal. This is accomplished by reversing the rotation of the crystallizer in a vertical or horizontal plane. The crystals are grown from melts, gaseous media, or solutions. The method is not too dissimilar to the "rocking crystallizers" used for single crystal production in the Western world. 35/

Interest in piezoelectric crystals in the Soviet Union has led to studies which have culminated in patents for growing crystals. P. G. Pozdnyakov and A. A. Shternberg grew ammonium phosphate in a pronounced unilateral direction by using a primer seed cut parallel to the edge of the pyramid. 36/ Another patent of theirs deals with the production of unilaterally growing crystals of ethylenediamine formate. Laminar seeds are placed in the pockets of the crystal carrier in such a manner that only the intersections of the rapidly growing facets are exposed. 37/ Ethylenediamine tartrate crystals were grown by Pozdnyakov from a solution containing a slight excess of ethylenediamine at a low supersaturation in order to avoid heterogeneities. A rate of growth of 5 to 8 millimeters per day was obtained at 45°C. with only 8° to 12° of undercooling. 38/ Pozdnyakov has also described the results of growing lithium sulfate and potassium tartrate crystals. By utilizing slow cooling or slow evaporation to get supersaturated solutions, he obtained crystals that are 65 grams in size for the former salt and 500 grams for the latter. 39/ 40/

Diffusion appears to be relatively unimportant as a controlling factor in crystal growth for highly stirred solutions if the solution is sufficiently supersaturated. It becomes the dominant force, however, when the solute has become sufficiently exhausted from the solution over a wide region. Ye. A. Arinshteyn expressed this mathematically:

$$V(t) = k (C^n(o,t) - C_o^n) \quad \text{Equation (1)}$$

where, $V(t)$ is the rate of growth at time (t) , C_o is the saturation concentration, n is the order of the process, and k is the kinetic coefficient. Equation (1) can be expressed as a series in powers of $t^{\frac{1}{2}}$ by:

$$V(t) = k (C_{\infty}^n - C_o^n) = (2nS \sqrt{k^2/\gamma D}) \cdot C_{\infty}^{n-1} (C_{\infty}^n - C_o^n) t^{\frac{1}{2}} + \dots \quad \text{Equation (2)}$$

where S is the density of the crystal. 41/

D. Ye. Ovsiyenko has showed that when crystals of galena are added to solutions of sodium chloride or sodium bromide, the supersaturation limit of each is markedly reduced because of the isomorphous nature of galena with the two salts. Melts of hydroquinone give similar results on metastability and orientation on various isomorphous materials. 42/

A kinetic study was made by I. V. Salli on the growth and the form of a crystal of the new phase that separates from a supersaturated solution. The rate of growth can be expressed by the equation:

$$dq/dt = 4\pi D \left[\Delta - (a/r) \right] \left[(1/x) + (1/r) r^2 \right]$$

Equation (3)

where, q is the amount of material diffused through a depleted zone around the crystal in time, t; D is the diffusion coefficient; $\Delta = C - C_r$ the difference in concentration in the solution, C, and at the crystal boundary, C_r ; r is the radius of the crystal; x is the thickness of the depleted zone; $a = 2\gamma M v / RT$, with γ the surface tension, M the molecular weight of the solute, and v the molecular volume of the solute. The effect of surface tension, γ , is found to be important only in the first stages of crystal growth. In quiescent solutions, the thickness of the depleted layer, x, can be considered constant. Therefore, the rate of growth can be shown to increase with a decrease in this constant thickness, x, and it passes through a maximum at a given size of crystal nucleus. 43/ 44/

The linear growth of the crystal boundary was measured by Danilov and V. I. Malkin for phenyl salicylate in the region of slight supercooling. They found the relation between the rate of growth and the supercooling to be exponential. The exponential curves coincided with the theoretical curves. 45/

Danilov and A. G. Pomgaybo studied the crystallization of highly purified sodium and potassium in both the presence and absence of their oxides. In each case, the maximum undercooling was reduced when even minute amounts of their oxides were present. 46/ The importance of impurities to act as crystallization centers was also dramatically shown by Danilov, O. D. Kozachkovskiy, and Ya. M. Labkovskiy through experiments with salol. Highly purified salol could be undercooled almost 100°C. without crystallization unless it was inoculated with salol seed crystals. 47/ Danilov and Kamenetskaya found that the slow cooling of highly purified mercury permitted undercooling by 18 to 21°C. without crystallization. However, additions of potassium to an amount of 0.05 per cent reduced the permissible supercooling by 7°C to 9°C. 48/

With a co-worker, Yu. A. Krishtal, Danilov demonstrated that the crystallization of an undercooled liquid can either be caused by extraneous impurities acting as crystallization centers or by spontaneous activity. This, of course, is dependent on the methods used to purify a substance. Three typical groups of substances can be distinguished as follows:

- (1) Salol represents Group I. It can be totally deactivated and then will not crystallize at any temperature without inoculation.
- (2) Ortho-chloronitrobenzene is a representative of Group II. When it is carefully purified and deactivated, it shows first crystallization centers at 20°C.

undercooling and cannot be undercooled more than 17°C . under any conditions. This represents a spontaneous crystallization limit.

- (3) Piperine is an example of Group III. This group must be cooled very slowly; otherwise it solidifies like a "glass." 48/ 49/

Danilov and Kamenetskaya's experiments on crystallization of azobenzene showed that undercooling before spontaneous crystallization could be markedly increased by the presence of soluble impurities, such as alcohol. 10/ Working with D. Ovsienko, Danilov extended this series of studies to the solidification of metals. In molten bismuth and lead, the presence of their respective oxides decreased the limits of metastability. 50/

Particles of insoluble materials in the presence of undercooled liquids can affect the degree of such undercooling. Danilov and Ovsienko found that hydroquinone could sustain 15° to 20°C . of undercooling. However, the undercooling was only 1.5° to 2.0°C . in the presence of crystals of calcite. 51/ In this same line of thought, Danilov and co-workers studied the formation of alpha-salol on rock salt particles. When alpha-salol is highly purified, it can withstand almost any degree of undercooling without the appearance of crystallization centers. In the presence of sodium chloride particles, crystallization centers appear in alpha-salol with a reasonable degree of undercooling. 52/

D. A. Petrov found the formation of a solid solution from a liquid solution involves two processes, namely the formation of new crystals and the alteration of the composition of the previous crystals. If the diffusion within the solid is very slow, the second process can be neglected and solidification takes place as if the solid phase was continuously being removed from the system. In this case, crystallization ends either at the temperature of the lower melting component or at the eutectic temperature. 53/

Intercrystalline liquation was studied by Petrov and L. A. Raykovskaya in aluminum alloys by a method of microhardness. These alloys ranged from 0 to 16 per cent magnesium and 0 to 10 per cent antimony. The hardness measurements were used to determine the degree of segregation in the alloy. The results proved that there was free diffusion in the liquid and no diffusion in the solid. 54/ The utilization of these experimental studies by Petrov and A. A. Bukhanova shows that greater homogeneity of metallic crystals from melts can be obtained by lowering extrusion velocity, improving intermixing in the melt, quickly cooling extended specimens, and avoiding a minimum of overheating in the melt. 55/

When mixtures of two or more components are crystallized, the additives are always redistributed between the liquid and solid phases. In non-equilibrium crystallizations, this redistribution causes macro- and micro-unevenness in additive distribution. Any large-scale unevenness is caused by incomplete liquid phase diffusion and appears by the liquid being gradually enriched in impurities that lower the melting point. These conclusions were based on studies of copper-aluminum, copper-zinc, tin-antimony, and aluminum-copper alloys made by Petrov and B. A. Kolachev. 56/

Ye. M. Savitskiy, V. F. Terekhova and A. V. Khlopov have studied the chromium recrystallization diagram. Electrolytic chromium tempered at 1300°C. in vacuo and at 1500°C. in hydrogen, with a volume reduction by deformation of 2.5 to 40 per cent, was studied photomicrographically, by X-ray spectrography, and microhardness determinations. Chromium recrystallization took place by simple crystal growth at the higher temperature and lower degree of deformation. However, at high deformations, it took place by formation and growth of new crystals. 57/ The recrystallization of titanium and its alloys was investigated by Savitskiy, M. A. Tylkina, and A. N. Turanskaya. They found that the recrystallization diagram can be divided into two parts. These two parts are the temperature regions where the α and β phases of titanium are stable. During the rolling of titanium, no recrystallization takes place. Recrystallization may be achieved by subsequent tempering or by further hot-working. 58/ The mechanical properties of titanium and its alloys were also determined by Savitskiy, Tylkina, and I. A. Tsyganova. They also constructed a recrystallization diagram for molybdenum from experimental data. 60/

N. A. Figurovskiy and T. A. Komarova found that the growth of crystals can be considered a free radical process in view of the chain mechanism and the crystallization process. The active centers where crystal growth occurs have a higher energy level than the other parts of the crystal surface. Separation of the solid phase from the solution begins at the active centers and is accompanied by the destruction of active centers and the simultaneous development of new ones. Potassium chloride crystallization has been used to study the above hypothesis. It has been shown that the rate of salt crystallization depends on the nature of the salt, the degree of supersaturation, the temperature, the impurities, and other factors such as the size of the vessel. The crystallization rate can be expressed by the temperature coefficient K_T :

$$K_T = \frac{1}{V_T} \cdot \frac{V(T + 10) - V_T}{(T + 10) - T}$$

wherein, $V(T + 10)$ and V_T are the crystallization rates at temperature $T + 10$ and T . An increase in temperature increases K_T . 61/

According to Danilov and Malkin, the growth of a crystal is accompanied by the fluctuation formation on its faces of two-dimensional nuclei and the subsequent spreading of these nuclei along the entire face of the crystal. If it is assumed that the nuclei spread along the entire face during a time that is short in comparison with the time of formation of new nuclei on the face, the linear speed of the crystals' growth is determined by the speed of origin of the two-dimensional nuclei. This theory may be expressed mathematically by the formula:

$$V = k_1 e^{-k_2/T_e} - k_3 / T^4$$

where, V is the linear speed of growth; T is the absolute temperature; T_e is the supercooling; k_2 is a constant denoting the energy of activation during the change of the molecule from liquid to solid.

$$k_3 = \frac{\pi^2 F \Gamma_m}{k Q_m}$$

in which p is the boundary tension on the edge of the two-dimensional nucleus; F is a unit of surface on which is distributed a monomolecular layer of one mole of substance; T is the melting point; and k is the Boltzmann constant. The first exponential in this equation deals with the mobility of the particles and decreases with increasing supercooling. The second exponential deals with the mechanism of crystal growth and increases with supercooling. Small supercooling was used on highly purified salol, and the theory as presented was experimentally confirmed. 45/

A. T. Grigor'yev has presented several excellent mathematical and theoretical papers on crystallization in binary and ternary systems:

- (1) The equation of the line of crystallization of the binary chemical compound in a binary system;
- (2) The equation of the surface of crystallization of this compound; 2nd
- (3) The equation for the surfaces of crystallization of the ternary chemical compound. 62/ 63/

A new method for determining the structural importance of the faces of crystals was reported by O. M. Ansheles. The method is based on the Kossel-Stranski theory of the growth of crystals. This simple solution assumes that the greatest distances are known over which the particles of the crystal interact practically with the particles of the liquid or gaseous phase. The theoretical part derives assemblies of physically possible crystal forms for simple, doubly idealized cases. In the practical part, the mechanism of a diamond crystal's growth is presented and the form of growth is derived. 64/

The absence of a lower boundary of miscibility is related to the formation of homogeneous mixed crystals that, from the point of view of thermodynamics, can be regarded as one phase. A formula was derived thermodynamically for such crystals by A. N. Kirgintsev. This formula relates the coefficient of equilibrium crystallization, D , with the activity of the micro- and macroelements in their saturated solutions and with the concentration of the macroelement in the mother liquor. Experimental data from the literature was used to examine the systems lanthanum fluoride - radium fluoride, lanthanum fluoride - thorium fluoride, and ammonium iodide - lead iodide, in detail. These systems confirm qualitatively the derived equations. 65/

G. G. Lemmleyn found the closing-up of a crack in a crystal by a saturated solution occurs spontaneously under isothermal conditions. This is due to the tendency of the crystal-solution capillary system, which has an excess surface per volume unit of the crack, to establish an energy equilibrium. The theory is developed to show that every concentration in a solution corresponds to a definite critical size of crystal particles that can exist in an equilibrium with that solution. 66/

V. M. Kravchenko has reported that three important and organically, very closely interrelated trends can be differentiated in the studies of the physical-chemical analysis of the equilibrium (liquid-crystalline) systems of various substances:

- (1) Topology, or qualitative study of the general geometric properties of the equilibrium-system diagram;
- (2) Metrics, i.e. quantitative study of the relationship between the elements of the diagram itself and the parameters characterizing the chemical system occurring in the system;
- (3) The science of the casual relationship between the topological and metrical characteristics of the diagram of the equilibrium of phases and the structure and properties of the system components.

These trends are treated through a series of developments which utilize an ideal phase-equilibrium diagram in a study of metallic, inorganic, and inorganic-organic systems. This development results in the conclusion that, for studying real crystal systems, ideal temperature-composition diagrams for crystallizing systems are as important as the concept of the ideal gas. 67/

A method was proposed by A. N. Liapunov and Ye. P. Kholmogortseva for determining the average growth rate of hydrargillite particles suspended in large numbers in a supersaturated aluminate solution, as a result of growth of the crystal faces. An equation was developed for determining the linear growth of the crystal faces to define total growth rate. The method may be applicable to other substances growing in supersaturated solutions. 68/

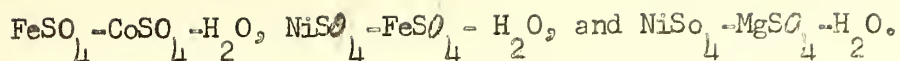
A. K. Skryabin mathematically and theoretically derived equations for the kinetics of the crystallization of solutions and melts with and without consideration of the medium's supersaturation and the gradient of concentration. The theoretically computed curves of the kinetics of crystallization for solutions and melts coincide with actual experimental data for supersaturated potash alum solution and for melts of zinc, lead, tin, and aluminum. 69/

The Soviet work on crystal growth is extensive and important. It is obvious, however, that much of the research is carried out to augment and supplement the need for practical application. Theoretical work on crystal growth thus tends to lag behind practical studies.

Crystals Systems

Ye. S. Makarov has collected, collated, and presented extensive data on the crystal chemistry of the actinide elements. The arrangements of atoms in crystals of actinides and their simplest compounds (chiefly thorium, uranium, plutonium and neptunium) are shown. A table is presented that shows the analogy between the crystal structure relationships of the actinides and the lanthanides and elements of the IVa, Va, and Via subgroups of the periodic system. 70/ Of the 117 references used by Makarov in compiling his book, only eight are Soviet. 71/ Once again, evidence is given of the present heavy dependence of the Soviets on Western work for the fundamental chemistry of the important nuclear fuel elements.

A series of good studies was conducted by G. I. Gorshteyn and N. I. Silant'yeva on the equilibrium distribution of isomorphic and isodimorphic components for various systems:



The coefficient of distribution, Dequil (iron, cobalt), is ideal at 20°C. and equal to 1.20. Other values vary, depending upon the ranges of concentration. A system of $\text{Co}(\text{NO}_3)_2 \cdot \text{Ni}(\text{NO}_3)_2 \cdot \text{H}_2\text{O}$ at 20°C. was also studied, and it was found to give solids of different crystal structures, since the two salts are not isomorphous. 72-74
Gorshteyn and Silant'yeva continued for another system, copper ammonium sulfate-zinc ammonium sulfate-water-at 20°C. utilizing radioactive isotopes of zinc (65) and copper (64) to determine the distribution coefficient. 75

The above important work resulted in Gorshteyn's development of the following conclusions, which have a bearing on the practical problems arising in connection with the industrial purification of inorganic salts. The study of the coefficient of distribution showed that it is nearly constant at any micro- or macroconcentration of the salts studied, as long as a solid phase with a definite structure remains constant. The experimentally established relations of the linear law of distribution are of considerable practical importance in problems relative to the fractionation of salts, specifically salts of rare-earth elements and other rare elements. Most of the work was developed by use of radioactive isotopes. 76

The preparation of high quality potash fertilizers from the Carpathian mines is difficult because of the multimineral nature of the raw material--sylvine, kainite, langbeinite, picromerite and polyhalite. A method has been developed by M. Ye. Pozin and M. I. Muratova for conversion of these minerals into potash fertilizers. A study of the crystallization of potassium chloride from kainite liquors showed that the quality of potassium chloride from kainite liquors showed that the quality of potassium chloride is independent of the time of cooling (below 45 minutes) and the temperature (above 30°C.) If the ratio of sylvite to kainite in the original ore is less than 0.3, good quality potassium chloride can be obtained by crystallization. 77 78

Ya. G. Goroshchenko has investigated the mutual solubility of the niobium oxide - sulfur trioxide - water system at 20°C. for a range of sulfur trioxide from 0 to 77 per cent. Two niobium sulfates were prepared-- $\text{Nb}_2\text{O}_5 \cdot \text{SO}_3$ and $\text{Nb}_2\text{O}_5 \cdot (\text{SO}_3)_2$; both are white solids, crystallizing either in needles or pseudo-cubes. 79 80 The crystal structure of the double salts of niobium and ammonium sulfate was also investigated. The three following salts were obtained: $\text{NH}_4\text{NbO}(\text{SO}_4)_2$, $(\text{NH}_4)_6\text{Nb}_2\text{O}_5(\text{SO}_4)_6$, and $(\text{NH}_4)_3\text{Nb}(\text{SO}_4)_4$. All are crystalline solids but decompose in water solutions. 80
Similarly, the double salts of tantalum and ammonium sulfate were studied. Two salts were obtained. Both are crystalline solids but decompose in water. 81

In the presence of various amounts of sodium hydroxide, curves were determined by M. I. Ravich and F. Ya. Borovaya for the phase diagrams of sodium sulfate - sodium chloride - water for pressures up to 200 atmospheres and temperatures to 600°C. 82

K. Ye. Mironov, Z. N. Pronina and S. A. Tokareva have made complete investigation of the phase diagrams of hydrogen peroxide - sodium perchlorate - water and hydrogen peroxide - lithium perchlorate - water. No peroxyhydrates of either sodium or lithium perchlorates were obtained; however, in the presence of hydrogen peroxides, various hydrates of each perchlorate were obtained. 83/

Ye. K. Akopov and A. G. Bergman found the system sodium chloride - potassium chloride - lithium sulfate to be a stable section of the complex mutual system. It is a simple triple system with an eutectic point at 426°C . and composition 24 per cent potassium chloride, 17 per cent sodium chloride, and 59 per cent lithium sulfate. 84/ The liquidus lithium fluoride and strontium fluoride were studied by Ye. I. Banashek and Berhman. The eutectic point was at 761°C . and 33.5 per cent strontium fluoride. For the chloride system, the eutectic point was at 487°C . and 54 per cent strontium chloride. 85/

The system hydrogen fluoride - iodine oxide - water was studied by N. S. Nikolayev and Yu. A. Buslayev. They observed the solid phases 0°C . were iodic acid and the double acid $2\text{HIO}_3\cdot 3\text{HF}$, a new compound. Compounds of the combined iodine-fluorine-oxide were not found at any concentration of hydrofluoric acid. 86/ N. K. Voskresenskaya and S. I. Berul found that the equilibrium in the system sodium nitrite - potassium nitrite - water at 70°C . shows a solid solution of sodium nitrite-potassium nitrite. At the eutectic point, this solution is more than 20 per cent potassium nitrite. 87/

While many other crystal systems were studied by Soviet scientists, those mentioned above show the wide diversity of such studies. The practical significance of the referenced systems is important to the expanding the Soviet chemical and metallurgical industries.

Habit Modification of Crystals

The presence of organic impurities in the mother liquor, even at low concentrations, materially influences crystal properties, habits of crystals, and crystallization. The interactions between impurities and growing crystal faces has received less study than has changes in habit. Such effects have important impact in crystallization.

Ye. N. Slavnova used spectrophotometric methods to elucidate the states of impurities (methylene blue) in barium and lead nitrate crystals. Lead nitrate takes up the dye mainly in the molecular state. This is, solid solutions of a special type are formed on the cube face growth pyramids. However, barium nitrate is entered by the dye as submicroscopic crystallites, giving a micro-heterogeneous system. When there are structural similarities between the components, the way in which impurities such as methylene blue enter the crystals is determined by the surface adsorption properties, the state of the impurity in the mother-liquor, and the tendency of the impurity to give adsorbed particles. 88/

Both crystals from metals and alloys and crystals from inorganic salts, viz. cadmium iodide, potassium perchlorate and others, under the presence of impurities in the melt, were studied by D. A. Petrov and B. A. Kolachev. They concluded that when mixtures of two or more components are crystallized, the additives (impurities) are redistributed between the liquid and solid phases.

This redistribution in non-equilibrium crystallizations causes macro- and micro-unevenness leads to two types of distribution: (1) layered distribution, and (2) substructures. 56/

The growth rate and shape of Rochelle salt (pure and plus borax) and potassium and ammonium aluminum alums (with added sodium carbonate) were investigated by A. V. Belyustin and V. F. Dvoryankin. They found the relative face growth rates can change with supersaturation in cases of the alums and pass through maxima and minima in Rochelle salt. The impurities used did not change the relative growth rates as functions of supersaturation but did alter the shape of the crystals. 89/

Clearly developed facial steps starting from the edges and corners and extending to the face centers are caused because the larger growth rates at corners and edges are much larger than on the faces. D. D. Saratovkin has discussed this type of growth in detail. These crystal forms are termed "skeletal." 90/ Ansheles has asserted the converse effect can also be observed. In other words, the face center layer growth rates can be much higher than the edge and corner ones. In this case, the faces become convex and take on antiskeletal shapes. 91/

A. F. Gorodetskiy and Saratovkin have made studies on cadmium iodide crystals. They stated, "Anti-skeletal growth forms on crystals, which arise from spiral growth on screw dislocations, can pass over to peculiar dendritic growth forms such as are also obtainable from skeletal forms. We term these layered dendrites, i.e. branching crystals with layered growth." Impurities play a strong part in producing large groups of screw dislocations, e.g., lead iodide growing in the presence of cadmium iodide. 92/

G. G. Lemmleyn and Ye. D. Dukova have proved naphthalene and p-toluidine, sublimed from a vapor phase, show spiral growths in the early stages of nucleation and crystallization, starting from the re-entrant angles of dendritic forms. Such dendrites are developed with variable types of branching. 93/ Other work of these two on growing a crystal of p-toluidine proved that the growth of elementary layers on the crystal face took place through the formation of a mobile adsorbed layer. 94/ Lemmleyn considered the properties of the boundary surfaces between crystals and solutions (or vapor) under conditions of equilibrium for both "positive" and "negative" crystals. The latter were crystals with closed cavities filled with liquid. 66/ He used hexamethylenetetramine in his experiments and showed that the "negative" crystals which are formed in saturated solutions over geological periods can be understood as nearly ideal equilibrium forms. 95/

A rather complete discussion of dendritic growth during crystallization, both of metals and of organic and inorganic compounds, has been presented by D. D. Saratovkin. Saratovkin's observations and conclusions are based on studies performed on crystallization with a stereoscopic microscope. The explanation of dendritic crystallization must be sought in an understanding of the role of impurities that are always present in crystallizing systems. 90/ A high percentage of Saratovkin's 127 references refer to Soviet studies. This shows the great interest and competence of Soviet scientists in this phase of crystallization, especially as applied to metal casting and solidification which dates back to the famous work of Chernov. 90/

V. I. Malkin observed that the transition to an acicular shape occurs when the rate of growth of a crystal of salol increases. When undercooling is decreased, the rate of growth decreases, and the growth of acicular crystals is replaced by the growth of crystals of regular shape. This transition was also observed in an ingot of crystallizing tin. 96/ This phenomenon was mathematically studied by G. P. Ivansov. He found that, under a given set of conditions, the product of the rate of crystal growth of acicular crystals and the radius of the tip of the crystal is a constant. 97/

The dislocation process that occurs during crystallization is not an accidental phenomenon. V. L. Indenborn found it is brought about by the irregular distribution of the temperature in a growing crystal. 98/

N. G. Zaytseva and A. M. Smirnova studied the rate of crystallization of hydrated tricalcium aluminate in the presence of surface-active substances, such as saponin and lignosulfonates. Small additions of surface-active substances increased the rate of crystal growth and repressed the speed of nuclei formations. 99/

Ye. S. Solov'yeva and Ye. Ye. Segalova stressed the importance of the structure of tricalcium aluminate since this compound forms part of all Portland cements. They found the induction period of structure formation (initial set) in which newly formed crystals appear is very short. The maximum stability of the crystal structure is reached when the anhydrous aluminate becomes completely hydrated. 100/

The stabilities of supersaturated solutions of barium iodate and lead iodide were studied by M. V. Tobin and S. I. Krasnova. The values of the maximum relative supersaturation that can be attained without bringing about spontaneous crystallization were determined. Additions of fuchsin, eosin, and atebirin to the supersaturated solutions showed that the maximum relative supersaturation of solutions of barium iodate and lead iodide was increased by adsorption of these materials on the solid crystalline phase. 101/ V. A. Mokiyevskiy studied epsom salt in the presence of borax, aniline, and methylene blue. The changes in the crystal form and the rate of crystallization were brought about by adsorption of the two organics on the growing crystal faces. 102/ N. P. Alekseyeva found that during the crystallization of potassium alum from solutions containing safranin, only surfaces grew on the crystals, while rhombododecahedron and octahedron surfaces were suppressed. 103/

The effect of xylan, dextrine, hemicelluloses, and resinous substances on the stabilization of calcium sulfate solutions was investigated by I. I. Korol'kov and Z. A. Tyagunova. All of these were effective in preventing precipitation of calcium sulfate and, of them, xylan was the most effective. 104/

S. V. Avakyan and N. F. Lashko studied, by microscopic means, the change in crystallization of the eutectic of potassium chloride and potassium dichromate in the presence of surface-active substances, such as agar-agar and sodium sulphonate. When agar-agar was used, the solution viscosity was increased and the crystal size of each component was reduced. They observed that sodium sulphonate does not change the viscosity of the solution, but it does decrease the size of the potassium chloride crystals. All tested surface active substances influence the process of eutectic crystallization by shifting the eutectic point. 105/

Crystallization Apparatus and Methods for Growing of Crystals

The optical glasses widely used in instruments have a major shortcoming in that they have low transparency outside the visible region. The useful wave length regions of the most common optical glasses are narrow (from about 0.33 to 2 microns) and therefore cannot be used in the ultraviolet and infrared regions. These shortcomings are especially acute in instruments below 0.2 micron. Only three materials have high constant transmissions in the short ultraviolet, i.e., the fluorides of sodium, lithium, and calcium. Of these, lithium fluoride has the most valuable combination of optical and mechanical properties for use in an optical material. Fluorite (calcium fluoride) has a high and uniform transmission over a wide wave length range of from 0.125 to 9.5 or 10 microns in the infrared.

Because of the scarcity of natural fluorite crystals of optical quality, I. V. Stepanov and P. P. Feofilov have developed and described a vacuum setup for growing fluorite at temperatures of 800° to 1500°C. Their method has been industrially adopted, and many optical components in the current instruments now are being prepared from artificial fluorite. 106/

M. A. Vasil'yeva has shown that lithium fluoride crystals are one of the best optical materials for use in vacuum spectrography of the far ultraviolet (below 250 millimicrons). They are also stable to atmospheric attack, very stable thermally, and have a degree of hardness only slightly inferior to that of fluorite. An industrial method for the vacuum growth of lithium fluoride crystals in graphite crucibles has been described by Vasil'yeva. 107/ Valuable optical crystals have been industrially produced by this method.

Scintillation counters have been widely used for recording nuclear radiations and for studying the emissions produced by the operation of various accelerators. The scintillator (crystalline, plastics, or liquid) is important, since this transforms the invisible nuclear radiation into a visible one. The transformation occurs when the radiation interacts with matter. Both organic and inorganic crystals, e.g., lithium bromide, cesium iodide, cadmium tungstate, etc., have been used as scintillators. L. M. Belyayev, B. V. Vitovskiy, and G. F. Dobrzanskiy have described methods for growing both organic and inorganic scintillators. 108/ Crystal scintillators have been used in scintillation counters for various purposes. The use of these counters has enabled Soviet physicists to solve completely new problems and to develop a new method of detecting nuclear radiations. 109/ 110/

The quality of semiconductor crystals must be uniform and predictable. The electrical properties of semiconductors are determined by the nature, contents, and distributions of impurities present or deliberately introduced and also by crystal structure perfection. Grain boundaries cause uncontrollable changes in the electrical properties, thus disturbing the electronic processes. The semiconducting materials must therefore be prepared from single crystals. Petrov and V. S. Zemskov have studied the growth of single crystals of germanium with uniform longitudinal impurity distributions of radioactive indium. They achieved this uniform distribution by melt crystallization with a constant feed into the melt. 111/

Mica is a valuable strategic raw material for insulators. K. V. Kapralov, Yu. V. Koritskiy, N. N. Sheftal', I. I. Yamzin, and M. S. Leyzerzon have discussed methods of growing and using large synthetic mica crystals. The synthetic mica-based ceramics are exceptionally resistant to thermal shock and are useful in equipment subject to variable heat loadings in gas turbine and jet engines and as coatings in the exhausts of supersonic jet aircraft. 112/ 113/

New piezoelectric materials are needed for various uses. Sorbitol hexaacetate has been shown to furnish good piezoelectric crystal plates for frequency stabilization. I. S. Rez and L. I. Tsinober have shown that it is possible to produce commercially single-crystal sorbitol hexaacetate from 96 per cent ethyl alcohol solutions. It should be noted, however, that the crystals are extremely sensitive to temperature variations and other disturbances during growth. 114/

Synthetic quartz crystals have been experimentally grown by V. P. Butuzov and L. V. Bryatov. They used experimental techniques developed by Western scientists, 400°C. and 1000-2000 atmospheres. Single-crystal seed plates were suspended at the top of the autoclave and broken pieces of unsuitable quartz were suspended at the bottom. 115/

Crystals of barium titanate and strontium titanate (and mixtures of the two) have been grown from solutions in fused salts by N. S. Novosil'tsev, A. L. Khodakov, M. L. Sholokhovitch, Ye. G. Fesenko, and O. P. Kramarov. These crystals can be grown best from molten potassium fluoride. The dielectric and ferroelectric properties of each crystalline compound are strongly dependent on their heat-treatment. 116/

Ya. V. Grechnyy has investigated the dependence of the probability of the nuclei formation of each phase upon the concentration of the liquid solution for the following two component organic systems: camphor - benzoic acid; camphor - naphthalene; camphor - ortho-chloronitrobenzene; camphor - paradibromobenzene. These substances have a tendency towards supercooling. It is concluded from the data that pure components have a minimum tendency towards supercooling, and that the tendency of the melt to supercool is further increased as its composition is removed from that of a pure component. 117/

B. D. Melnik has shown that salts can be subdivided into two groups based on solubility changes with the temperature:

- (1) The solubility first rises with the temperature, then drops to practically zero at some temperature with the formation of either anhydrous salts or salts containing only a little water of crystallization, e.g., the sulfates of sodium, potassium, magnesium, manganese, and nickel.
- (2) The solubility increases with the temperature, e.g., most halide salts and ammonium compounds. An elevated temperature improves crystallization separation techniques. Commercial methods using these principles are described. 118/

In the crystallization of sugar from its aqueous solutions, M. B. Yarmolinskiy found that the amount of crystals increases with the supersaturation coefficient. Stirring a supersaturated solution of sugar promotes the formation of seed crystals and thus improves the fineness of the granulated product. 119/

A. Mircev and K. Sandera have conducted studies on crystallization and product centrifugation of sugar from massecuite in the presence of added salts. They found in a plant-scale experiment that sodium sulfite and calcium chloride improved crystallization, reduced the viscosity of intracrystalline molasses, and improved centrifugation. Calcium nitrate and aluminum sulfate gave poor results. 120/

The effect of ultrasonics of the kinetics of crystallization on thymol was studied by A. P. Kapustin. It was shown that ultrasonics produced "ingots" which are more uniform and which are composed of smaller crystals. Ultrasonics (720 kilocycles/second) modified the character of eutectic and "orthotropic" crystallization, i.e., growth perpendicular to the plane of formation of crystals. It also suppressed columnar structures. 121/ The crystallization of benzophenone (I), o-chloronitrobenzene (II), thymol (III), piperonal (IV), salol (V), ammonium chloride (VI), and condensed milk (VII) in the presence of ultrasonics was observed with a polarization microscope equipped with a camera. In the case of the first group (I, III, IV) the sound caused a splitting-off of newly formed crystallites, directly proportional to the intensity of the sound. In the second group (II, V, VI, VII), ultrasound caused the breaking of growing crystallites, which served as new centers of crystallization. In both groups the rate of crystallization was several hundred times that found under ordinary conditions. 122/

Kapustin also has studied the effect of ultrasound in the range of 720 kilocycles to 6 megacycles per second on the rate of crystallization of solutions and alloys. The increase in the rate was due to the formation of a larger number of crystallization centers. The rate was increased by a factor of one hundred. The ultrasound waves are effective only if the energy reaches a certain level. No further effect is observed above this limiting level. 123/ Alum crystals were grown by Kapustin and V. Ye. Kavalyunayte in identical experiments. One was grown with no ultrasound and one was grown with the application of an ultrasonic field of 2 megacycles per second and intensity of 0.2 watts per square centimeter. The linear dimensions of the crystals grew at double the rate when the ultrasound was applied. 124/

The term "texture" can be used to describe a certain preferred orientation of crystals in a crystal aggregate. Interest in texture is important since the physical properties of a crystalline substance depend not only upon the internal lattice atomic arrangement but also upon the size of the individual crystallites and their orientation. By using ultrasonics, Kapustin was able to obtain a special texture in molten potassium sodium tartrate in the form of regularly arranged planes, subject to the frequency of the ultrasound. 125/

N. T. Gudtsov has observed the effect of ultrasound upon the phenomenon of orthotropism of organic substances. The macrostructure of a crystalline aggregate is characterized by the presence of several zones of varied structure. The external zone consists of acicular crystals, in which the direction of growth is perpendicular to the walls of the mold. In metals, parallel fiber crystal orientation leads to weakened loads and sometimes to disintegration (or deep crevices) during rolling. The complete elimination or minimization of acicular crystals was achieved in organic substances by applying ultrasound. Thus, the process may be used in casting techniques to obtain a more uniform, fine crystalline structure. 126/

Many investigators have sought new methods for the control of processes involving phase changes. Of these methods ultrasound has been shown to increase the rate of crystallization and to produce finer crystal aggregate structures. In a study made by I. G. Polototskiy, T. Ya. Beniyeva, and Z. L. Khodov, ultrasound was applied to a melt of ortho-chlorobenzene and to molten zinc, cadmium, and tin. It was proved that centers of crystallization take place in the nodes of the standing ultrasonic waves and in finely dispersed admixtures. With these increased numbers of crystallization centers, fine aggregates of crystals, i.e., fine grained metal structures, were obtained. 127/ This phenomenon is of great practical importance.

The formation of crystals in a supercooled liquid may be forced or spontaneous. Ortho-chloronitrophenol was studied by Kapustin to show that spontaneous crystallization could take place in highly purified samples at a supercooling of 18°C. Under the influence of ultrasound, the emergence of the first crystallization center was highly accelerated. 128/

Various theories have been advanced on the increased rate of crystallization under the influence of ultrasonics. B. V. Vitovskiy states that the use of sonic vibrations removes the layer of depleted solution adhering to the face of a crystal and exposes the crystal face to fresh supersaturated solution. Thus, the growth of the crystal face is increased. 129/

An apparatus was designed and built according to Verneuil's technique for growth of artificial rubies. This apparatus was used by D. A. Petrov and Yu. M. Shashkov to grow silicon crystals. 130/

A new type of crystallizer for naphthalene fractions was designed by V. Ye. Privalov and has been put into commercial operation. Essentially, it consists of tubes, which can either be cooled by cold water or heated by steam. The hot naphthalene fractions are put into the tubes, crystallized by cooling and removed by heating. Production of this type of tube crystallizer is much higher than that of drum coolers. 131/ The formulas for designing drum and mechanical crystallizers were presented by V. M. Tamarin and have met commercial requirements. 132/ L. N. Matusevich has developed a mechanical laboratory crystallizer to determine the effect of stirring intensity on the process of crystallization and the physical phenomena connected therewith. Investigations on its use have shown its value in laboratory studies. 133/

As early as 1947, it was observed that a considerable increase in the velocity of crystal orientation was achieved under the influence of ionizing radiation. M. L. Bookin and A. A. Semerchan thoroughly investigated this phenomenon and showed that, under the action of electron radiation, aluminum foil reacted with the oxygen of the air to give aluminum oxide crystals. The size of the crystals produced depended on the dose of irradiation. It is postulated that this process can be utilized for the production of inexpensive tools of high quality from aluminum oxide for metal cutting. 134/

Electric (electronic) processes in semiconductors depend very strongly on negligible amounts of impurities and structural defects. Fractional recrystallization (zone refining) is one method of obtaining pure germanium and silicon. However, since diffusion proceeds slowly in the solid phase, the impurities vary as the ingot grows in the melt. By continuously supplying melt for special

purposes, D. A. Petrov and B. A. Kolachev showed the same result can be accomplished as in the pulling process. Crystals can be obtained of a constant composition along the length of the solid. In order to estimate the effectiveness of purification a distribution coefficient, k , is used:

$$k = c_s/c_m$$

where, c_s is the concentration of impurities in the solid and c_m in the melt. If k is constant, then one obtains:

$$y = kx_0(1 - m_s)^{k-1}$$

where, y is the impurity content in the growing crystal on the crystallizing front, x_0 is the initial melt composition, and m_s is the mass of the solid ingot already formed from the melt. Experimental results on various metals, including germanium and silicon, have shown the value of the technique for purification to allowable tolerances for semiconductors. 135/

F. M. Kutsakov has devoted a study to inducing crystallization in chemical reactions by rubbing. Eleven different solutions, to which appropriate precipitating solutions were added, were placed in test tubes or on plates of various materials. A variety of materials was used to rub the solution containing materials. The rate of precipitation (crystallization) was increased by this technique. 136/

From the references reviewed it can be concluded that:

- (1) The Soviet research uses standard techniques;
- (2) The investigations lead to steady progress and development of practical apparatus and methods;
- (3) No outstanding results were reported, which were not already available to the Western world;
- (4) Soviet authors write as if each paper (report) presents new outstanding results. (This may be due to awkward translations.)

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EXTRACTION

The Soviet literature on extraction is substantial in quantity, but not extensive. Its quality is generally below average by Western standards, although recent papers indicate considerable improvement and a growing trend toward originality, particularly in the treatment of theory. Emphasis on process development appears to be increasing but reduction of new processes to plant practice is retarded by limited capacity for original equipment design. Soviet work in extraction will be discussed under four general headings: theory and calculation methods, equilibrium studies, processes, and equipment.

Theory and Calculation Methods

Soviet scientists appear to have done little original work on extraction theory and calculation methods. Until very recently, papers on these subjects have been virtually absent from their publications. As confirmed by their own review articles, the Soviets are familiar with Western developments in extraction technology and have generally accepted these, offering only occasional improvements and empirical extensions. 1/2/ For example, vector projections were suggested as convenient for graphical interpretation of phase diagrams. 3/ N. I. Smirnov and colleagues have studied droplet motion in liquid media under laminar, transition and turbulent flow conditions; they developed a series of empirical equations pertinent to extraction hydraulics. 4/5/6/ V. V. Kafarov has presented a generalized correlation of flooding-velocity data in packed towers. 7/

However, the Soviet literature since 1957 shows marked improvement in quality and a shift in emphasis toward more fundamental work. Leading this trend is V. V. Kafarov of the Moscow Technological Institute imeni D. I. Mendeleev, who is seeking to extend the turbulent-layer theory (see discussion of adsorption) to extraction and other mass-transfer processes. 2/ 8/ 9/ 10/ In his latest paper, Kafarov applies this approach to the interpretation of experimental data on phase contact between mutually insoluble liquids with mechanical stirring. 11/

Other Soviet workers are beginning to follow Kafarov's lead. For example, G. K. Goncharenko and A. P. Gotlinskaya analyzed results from over 100 liquid-liquid extraction experiments with various substances; they found that mass-transfer coefficients under fully developed turbulence are independent of distribution coefficients and concluded that mass-transfer rates are then controlled by an interface resolution reaction involving breakdown of a solvate shell in one solvent and formation of a new shell in the other. 12/ V. A. Marinin is investigating the activation energy of diffusion processes by means of viscosity measurements. 13/ From theoretical considerations of bond energies and molecular motion, G. M. Panchenkov has developed a generalized equation for the temperature-dependence of diffusion coefficients in liquids:

$$\log D = \log A - \frac{e_0}{RT} + \alpha \log T$$

where e_0 is the energy of a single liquid molecular bond and A and α are constants derived from physical properties. 14/ By departing from the customary "energy of activation" approach to diffusion, which would lead to only the first two terms on

the right side of his equation, Panchenkov may have made a significant new contribution. However, experimental data to support his equation are still in the process of collection. 14/ 15/

Over a period of years, I. L. Krupatkin* has been conducting an extensive study of phase stability and layer formation in liquids. 16-34/ Much of this work has consisted of data collection and empirical correlation on binary and ternary systems composed of various organic acids, amines, alcohols and water; some of these involve compound or complex formation, and others do not. Krupatkin classifies ternary systems into three types, depending upon whether one, two, or all three of the binary systems are completely miscible. He suggests a "law of inverted similarity" which relates the shapes of the binary separability isotherms in a given system and permits limited qualitative estimation of ternary phase behavior from binary system data. 23/ His theory for the mechanism of liquid stratification involves a three-stage polythermic process of liquid solution decomposition, followed by a diffusional phase-formation process. 27/ Krupatkin has suggested and applied a method for studying chemical interaction in multiphase liquid systems by means of two solvents of different volatilities. 22/ 29/ 30/ His latest papers depend less on empirical analysis and more on thermodynamic reasoning, seeking to show that metastable separations are governed by the same principles which apply to stable phase equilibria. 32/ 33/ 34/ Krupatkin's extensive accumulation of data and experience in this area makes him a leading Soviet specialist on liquid separations. His papers indicate that he is not directly interested in extraction applications, but his findings could form the basis for significant work by others.

Recent research in the USSR on extraction theory is not entirely original, since admittedly much of it stems from current discussions in the Western literature; but there is evidence of the present sophistication of Soviet specialists in this area and of their probable capability to contribute significantly now and in the future. Thus, the Soviet attitude toward extraction fundamentals, backward and imitative as recently in 1956, appears to have come of age rather rapidly.

Equilibrium Studies

Recent Soviet research on extraction equilibria is summarized in Table on page 107. With only a few exceptions, water is involved as one of the solvents. No general pattern is evident from this work, but some of the systems studied suggest orientation toward the pharmaceutical industry. 17/ 24/ 26/ 30/ 31/ 51/ 52/ The data on fused salt systems may have metallurgical applications. 62-65/

Until sometime in 1957, Krupatkin was at the Cherkassy Pedagogical and Teacher's Institute. 16/ 30/ His most recent papers place him at the Yaroslavl Technological Institute 32/ or the Ivanovo Chemical Technology Institute, Chair of Inorganic Chemistry. 33/ 34/

During the past two years several papers reporting on systems related to nuclear energy developments have appeared, suggesting that some of the information in this area accumulated in the Soviet Union is now being released for publication. 57/ 58/ 59/ 61/ However, the data disclosed in these papers appear rather limited.

Soviet interest in extraction equilibria seems to be concentrated at a relatively small number of research institutions. For example, eleven of the entrees in Table I are from the work of I. L. Krupatkin at the Cherkassy Pedagogical and Teacher's Institute. 16/ 17/ 19/ 24/ 26/ 30/ 31/ 52/ Five references are papers by S. Sh. Byk and colleagues at the Scientific Research Institute on Synthetic Alcohols and Organic Products in Moscow. 37/ 45/ 46/ 47/ 48/ Five others are from the Leningrad State University imeni A. A. Zhdanov. 38/ 40/ 49/ 50/ 56/

Aleksey Vasil'yevich Storonkin at Leningrad State University is the leading Soviet specialist in thermodynamic analysis of phase equilibria; his principal associates are N. P. Markuzin, A. G. Morachevskiy and M. P. Susarev. They have published a series of papers deriving mathematical relationships for thermodynamic equilibrium in multicomponent systems. 71-76/ Storonkin has coined the term "isotherm-isobar" for the family of curves (or surface) expressing the possible temperatures and pressures of two-phase coexistence in ternary systems; he has discussed theoretically possible shapes for isotherm-isobars and demonstrated their applicability to solubility studies. 74/ He has also suggested some semi-quantitative rules for predicting ternary phase relationships from data on binary mixtures containing common components. 72/ Storonkin's thermodynamic reasoning follows established conventional lines, but it is rigorous and thorough, as evidenced by his generalized mathematical treatment of open and closed systems involving n-components and r-phases. 76/ In applying thermodynamic analysis to experimental data, Storonkin appears interested primarily in vapor-liquid equilibria. However, several of his papers in this area also discuss solution effects pertinent to extraction. 49/ 50/ 56/ 77/ 78/

At the M. I. Kalinin Polytechnic Institute in Leningrad, A. F. Alabyshev and M. F. Lantratov have been studying the thermodynamics of fused salts and molten metals; 79-85/ Morachevskiy has also been associated with some of this work 83-85/ Their experimental technique is electrochemical, evaluating the activity of solution components from potential measurements on concentration cells, and the quality of the work appears to be excellent. The fused salt studies have covered the activity of lead, cadmium and zinc chlorides in sodium potassium and barium chloride melts. 79-81/ The molten metal systems investigated include sodium - cadmium, 83/ lead - sodium - cadmium, 84/ and zinc - antimony - tin 82/; in this work entropy, free energy, and enthalpy effects were calculated, in addition to activity coefficients. A recent paper by these authors reviews and summarizes various Soviet studies of liquid metal thermodynamics. 85/ In view of current Western work on liquid metal extraction processes, both for general metallurgical applications and for processing of nuclear fuels and fission products, Soviet activity and competence in this area may be quite significant.

A. M. Rozen and L. P. Khorkhorina have discussed the thermodynamics of the equilibrium distribution of uranyl nitrate and nitric acid in the extraction of aqueous solutions with mixtures of tributyl phosphate (TBP) and organic solvents. 86/ 87/ They analyzed their own data 61/ and used some of the concepts suggested by Fomin and Mayorova, 60/ but they referred extensively to British literature (primarily to the work of H.A.C. McKay, et. al., Trans. Faraday Society). They

concluded that in these systems distribution constants depend strongly on the non-ideality of TBP - organic solvent solutions, which tends to promote extraction. Rozen and Khorkhorina derived a general equation for calculating the combined distribution of uranyl nitrate and nitric acid between the aqueous and organic phases; 87/ however, use of this equation requires a knowledge of distribution and activity coefficients. Rozen also studied the effects of various nitrates as salting-out agents, suggesting an equation for correlating these effects in terms of conventional solution parameters. 86/ The line of reasoning followed in these papers parallels that customarily used in the Western literature, offering little in the way of theory development or extension. However, the work of Rozen and Khorkhorina demonstrates competence for employing and understanding contemporary reasoning and knowledge in this area.

Extraction Processes

Recent literature points to increasing emphasis on extraction process development in the Soviet Union. Much of this effort centers on methods for preparing nuclear fuels and separating fission products and on processes related to metallurgical industries. However, interest in organic processes is also evident, as well as noteworthy activity in the application of extraction techniques to analytical chemistry.

A leading Soviet specialist in the extraction of uranium and fission products is V. M. Vdovenko at the Institute of Radium imeni V. G. Khlopin. His publications are all very recent (1957-1959) and cover both process and analytical applications 88-90/ as well as basic chemistry. In the latter category, Vdovenko and colleagues have reported extensively on solubility relationships for uranyl nitrate in dibutyl and diethyl ethers; 68-70, 91/ on heats of solution in these systems; 92/ on the distribution of uranyl nitrate and other metal nitrates between water and various ethers and esters; 93-95/ and on uranyl chloride complexes in organic solvents. 96/ Studies such as these apparently provide the fundamental data for Vdovenko's extraction process work. For example, his systematic investigation of the distribution of uranyl, neptunyl, and plutonyl nitrates between aqueous solutions and oxygen-containing organic solvents led to the development of a two-cycle extraction process for separating and recovering practically pure uranium and plutonium from fission products, using a non-explosive mixture of dibutyl ether and carbon tetrachloride as the solvent. 97-99/ In another process study, still in the laboratory stage, Vdovenko and L. N. Lazarev are investigating the extraction of uranyl acetate with aniline. 100/

V. D. Nikol'skiy and V. S. Shmidt have reported on the extraction of ruthenium in the form of nitrosonitrates from aqueous solutions of irradiated uranium. 101/ I. V. Shilin, A. S. Solovkin and associates are studying tributyl phosphate extraction of perchloric acid, with the aim of developing a process for recovering and concentrating uranium as uranyl perchlorate. 102/ 103/

Recent Soviet publications on metallurgical extraction processes show a wide range of interests, suggesting an extensive program of mineral resources development. Some of this work is directed at up-grading waste materials. For example, V. G. Agyenkov and Ya. Ya. Miklun have reported on methods of improving the extraction of gold from sulfide concentrates. 104/ L. L. Chermak has investigated the distribution of cobalt in molten converter slag; 105/ this work resulted in a patented process for extracting nickel and cobalt from slags by means of cadmium sulfide mat. 106/ Ya. Z. Malkin and associates developed a method for extracting tellurium from lead production wastes. 107/ V. G. Kovyrrshin and V. K. Apollonov have reported successful laboratory and pilot plant studies on the extraction of calcium molybdate mother liquors for

molybdenum and rhenium recovery. 108/ A. I. Mikheyeva and V. B. Aleskovskiy have proposed a novel scheme for recovering copper from dilute solutions by countercurrent contact with alumino-silicate particles carrying adsorbed ammonia. 109/ Another unusual technique reported recently in the Soviet literature is a "mercury trap" method for separating rare metals (gallium, indium and thallium) by extracting them from water solution with zinc amalgam and then electrolyzing the amalgam. 110/

In the area of new but more conventional processes are flotation studies of indium ore recovery by O. A. Shubina and L. I. Chechulina. 111/ and of phosphate rock enrichment by L. I. Stremovskiy 112/ Extraction of alumina from shale ash with nitric acid has been investigated by R. I. Agaladze and I. M. Arazashvili. 113/ Ye. A. Bylina and associates have experimented with carbon tetrachloride extraction of germanium from coal. 114/ A. M. Rozen and four colleagues have patented a process for extraction separation of metals from aqueous solution by means of fatty acids dissolved in kerosene. 115/ Most of the above process literature appears to result from laboratory investigations, suggesting limited Soviet capability for carrying new extraction process developments into successful plant operation.

Relatively few Soviet studies of organic extraction processes have been published recently, and these do not emphasize any particular area of interest. However, they do show more of a tendency toward experimentation on a pilot plant scale than indicated in the metallurgical extraction work. A. V. Mazov and colleagues reviewed the practice of extractive separation of coal tar fractions in the USSR and reported pilot plant results on a new process for phenol recovery using aqueous methanol solutions. 116/ V. N. Kozlov and G. A. Tokareva studied the extraction of formic, acetic, propionic, and butyric acid mixtures in a ten-stage counter-current apparatus, using diethyl ether, ethyl acetate, and butyl acetate as solvents. 117/

Several papers proposing new extraction processes for the petroleum industry have appeared. For example, M. A. Kapelyushnikov and T. P. Zhuze have suggested the use of compressed hydrocarbon gases at 100-150 atmospheres pressure for extracting resin from crude oil. 118/ B. K. Marushkin and colleagues studied the effects of recycle on the extraction of crude oil with phenol. 119/ A. K. Seleznev proposed the use of beta-chlorodiethyl ether and dichloroethane mixtures for solvent dewaxing of lubricating oils. 120/ V. B. Kogan and associates reported that ethylene glycol is an effective solvent for extracting alcohols from mixtures with hydrocarbons. 121/

A number of Soviet chemists have become specialists in the application of extraction techniques to chemical analysis. These include V. M. Vdovenko at the Institute of Radium imeni V. G. Khlopin, whose work is cited above; 88-96/ I. P. Alimarin and I. M. Gibalo at Moscow State University imeni M. V. Lomonosov; and V. I. Kuznetsov and K. V. Troitskiy at the Institute of Geochemistry and Analytical Chemistry imeni V. I. Vernadskiy.

Alimarin and Gibalo have developed methods for extracting niobium, tantalum, and tungsten from hydroxyquinolate complexes with isoamyl alcohol and chloroform 122/ and from cupferronates with chloroform, ethyl acetate, or diethyl ether. 123/ They have also devised a technique for separating beryllium from other metals by extracting a mixture of acetylacetone complexes with carbon tetrachloride. 124/ Recently Alimarin reported a new method for isolating neptunium from solutions containing uranium and plutonium by extraction with nitroso-naphthol dissolved in butyl alcohol. 125/

Kuznetsov, who publishes actively in various areas of analytical chemistry, has made an extensive review of the theory and practice of extractional separation of metals. 126/ His own contributions include a procedure for extracting vanadium from acid solutions with ketones, 127/ and a new technique for the rapid separation of various groups of metals by extraction with fused low-melting organic substances. 128/ Troitskiy has been studying niobium extraction from thiocyanate complexes by organic solvents, using radioactive tracer techniques. 129/

Other recent Soviet contributions worthy of mention in this area are discussions of the thermodynamics of analytical extractions by N. P. Komar 130/ and by I. M. Korenman and F. R. Sheyanova; 131/ 132/ studies by F. G. Zharovskiy of molybdenum and tungsten extraction from chloride complexes; 133/ and investigations of cerium extraction by V. M. Klinayev and M. M. Senyavin 134/ and by A. V. Nikolayev and associates. 135/

Extraction equipment

Corresponding to growing Soviet interest in extraction processes, recent literature shows increased activity in the study of extraction equipment. Much of this work is concerned with apparatus and operating techniques originated outside of the Soviet Union, suggesting that Soviet specialists in this area are currently engaged in "catching up" with foreign advances and that their own capacity for original equipment design in support of new process developments still is rather limited. For example, a review of operating characteristics of various extraction devices, published in 1958 by N. I. Gel'perin and A. G. Liakumovich, contains 68 references, of which only 9 are Soviet, 46 are English and ten American. 136/ A paper by S. M. Karpacheva and co-workers reports operating tests on a pulse column modeled after an American patent. 137/ G. P. Piperskikh and Ye. R. Valashels describe and analyze the performance of three centrifugal extractors, all of non-Soviet design. 138/ A recent paper by S. Z. Kagan and associates, reporting a series of tests on rotary disc extractors, cites 26 references, of which only six are Soviet. 139/

However, some noteworthy new equipment suggestions have appeared in the Soviet literature. Based on a fundamental study of liquid dispersion in flow through nozzles, 140/ N. I. Gel'perin has designed an extraction column employing injectors at each end to insure fine dispersion of both feed solution and solvent. An experimental model of this injector column has been tested with a water- uranyl nitrate- tributyl phosphate system, 141/ and is claimed to be twice as effective as a plate or packed column of conventional design. 142/ S. M. Karpacheva and co-workers suggest that extraction efficiency in this type of column can be increased further by introducing multiple jets. 143/ Gel'perin has also devised a new technique for laboratory extractions whereby a volatile solvent is introduced as vapor into a column, achieving improved mass transfer by solvent condensation. 144/ He is currently studying the operation of an experimental column containing alternate mixing stages and sections packed with Razchig rings. 145/

V. V. Kafarov, whose contributions to extraction theory are discussed above, has also studied the effectiveness of liquid jets as extractors. 146/ Recently Kafarov patented a new column design consisting of pump-mixers and annular settling chambers arranged alternately. 147/ Another scheme for jet column operation, whereby several jets for feeding fresh solvent are spaced along the height of the column, has been patented by I. V. Filippov. 148/ As an alternate to using liquid jets, N. P. Galkin and associates have proposed the introduction of an air stream near the bottom of plate columns to improve light-phase dispersion. 149/

Three recent papers by staff members of the Moscow Chemical Machine Building Institute infer strong emphasis there on extraction equipment development. A. N. Planovskiy and S. N. Bulatov have studied drop size and formation frequency in relation to hydrodynamic conditions in sieve-plate extraction columns, using both electromechanical and oscillographic methods. 150/ A. K. Skryabin has compared theoretically and experimentally the relative merits of jet dispersion of the light and the heavy phases in extraction columns. 151/ I. I. Salamatov and G. M. Veksler have designed a new continuous rotary extractor that operates without pumps and achieves efficient and rapid extractions. 152/

It is interesting to note that nearly all of the significant current Soviet work on extraction equipment is at institutions located in Moscow. The last three papers mentioned are from the Moscow Chemical Machine Building Institute. Gel'perin is at the M. V. Lomonosov Institute of Fine Chemical Technology, as of 1958. 142/ Kafarov is at the Moscow Technological Institute imeni D. I. Mendeleev, as of 1959. 11/ 147/ It thus appears that a concerted effort may be underway to hasten correction of the Soviet weakness in this area of chemical technology.

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Appendix A

Key to Journal Abbreviations

Dokl. AN SSSR

Doklady Akademii Nauk SSSR

Dopovidi AN UkrSSR

Dopovidi Akademii Nauk Ukrayins'koyi Radyans'koyi Respubliki

Izv. AN BSSR

Izvestia Akademii Nauk Belorusskoy Sovetskoy Sotsialisticheskoy Respubliki

Izv. AN SSSR. Otd. Khim. N.

Izvestiya Akademii Nauk SSSR. Otdeleniye Khimicheskikh Nauk

Izv. AN SSSR. Otd. Tekhn. N.

Izvestiya Akademii Nauk SSSR. Otdeleniye Tekhnicheskoy Nauk

Izv. AN SSSR. Ser. Fizicheskaya

Izvestiya Akademii Nauk SSSR. Seriya Fizicheskaya

Izv. Sektora Fiz.--Khim. Analiza IONKh, AN SSSR

Izvestiya Sektora Fiziko--Khimicheskogo Analiza, Akademii Nauk SSSR,
Institut Obshchey i Neorganicheskoy Khimii imeni N. S. Kurnakova

J. Appl. Chem. (USSR)

Journal of Applied Chemistry (USSR)

J. Phys. Chem. (USSR)

Journal of Physical Chemistry (USSR)

J. Phys. (USSR)

Journal of Physics (Academy of Sciences USSR) (Continuation of Technical Physics
of the USSR. Apparently suspended with 11, No. 5, 1947)

Khim. Mashinostroyeniye

Khimicheskoe Mashinostroyeniye

Khim. Nauka i Prom-st'

Khimicheskaya Nauka i Promyshlennost'

Khim. Prom-st'

Khimicheskaya Promyshlennost'

Khimiya i Khim. Tekhnol. Sokr. Perev. iz In. Period. Lit

Khimiya i Khimicheskaya Tekhnologiya. Sokrashchennyye Perevody iz Inostrannoy
Periodicheskoy Literatury

Khimiya i Tekhnol. Topliv i Masel

Khimiya i Tekhnologiya Topliv i Masel

Kolloidn. Zh.
Kolloidnyy Zhurnal

Metody Issledovaniya Struktury Vysokodispers. i Poristyykh Tel, AN SSSR,
Tr. Soveshchaniya
Metody Issledovaniya Struktury Vysokodispersnykh i Poristyykh Tel, Akademiya
Nauk, Trudy Soveshchaniya

Quart. Revs. (London)
Quarterly Reviews (London)

Ref. Zh. Khimiya
Referativnyy Zhurnal - Khimiya

Reports at the First Conference on Crystal Growth, 5-10 March 1956
Growth of Crystals (Rost Kristallov); Reports at the First Conference on
Crystal Growth, 5-10 March 1956, in English translation, Consultants
Bureau, Inc., New York, New York

Sakharnaya Prom-st'
Sakharnaya Promyshlennost'

Sb. Fiz.-Matem. Fak. i N.-I. In-ta Fiz. (Odessk. Un-t)
Sbornik Fiziko-Matematicheskogo Fakul'teta i Nauchno-Issledovatel'skogo
Instituta Fiziki (Odesskiy Gosudarstvennyy Universitet imeni I. I.
Mechnikova)

Tr. In-ta Chernoy Metallurgii. AN UkrSSR
Trudy Instituta Chernoy Metallurgii, Akademiya Nauk Ukrainskoy SSR

Tr. In-ta Fiziki. AN UkrSSR
Trudy Instituta Fiziki, Akademiya Nauk Ukrainskoy SSR

Tr. In-ta Kristallogr. AN SSSR
Trudy Instituta Kristallografii, Akademiya Nauk SSSR

Tr. Komis. po Analit. Khimii. AN SSSR
Trudy Komissi po Analiticheskoy Khimii, Akademiya Nauk SSSR, Institut
Geokhimii i Analiticheskoy Khimii imeni V. I. Vernadskogo

Tr. Leningr. Tekhnol. In-ta im. Lensovet
Trudy Leningradskogo Tekhnologicheskogo Instituta imeni Lensovet

Tr. Mosk. Khim.-Tekhnol. In-ta im. D. I. Mendeleyeva
Trudy Moskovskogo Ordena Lenina Khimiko-Tekhnologicheskogo Instituta imeni
D. I. Mendeleyeva

Tr. Radievogo In-ta im. V. G. Khlopina
Trudy Radievogo Instituta imeni V. G. Khlopina

Tr. Vses. N.-I. In-t Sakharn. Prom-sti
Trudy Vsesoyuznyy Nauchno-Issledovatel'skiy Institut Sakharnoy Promyshlennosti

Uch. Zap. Kishinevsk. Un-t
Uchenyye Zapiski, Kishinevskiy Gosudarstvennyy Universitet

Uch. Zap. LGU
Uchenyye Zapiski Leningradskogo Gosudarstvennogo Universiteta imeni A. A.
Zhdanova

Uch. Zap. MGU
Uchenyye Zapiski, Moskovskiy Ordena Lenina Gosudarstvennyy Universitet
imeni M. V. Lomonosova

Ukr. Khim. Zh
Ukrainskiy Khimicheskiy Zhurnal

Vestn. AN SSSR
Vestnik Akademii Nauk

Vestn. Leningr. Un-ta, Ser. Fiz i Khimii
Vestnik Leningradskogo Universiteta, Seriya Fiziki i Khimii

Zh. Analit. Khimii
Zhurnal Analiticheskoy Khimii

Zh. Eksperim. i Teor. Fiz.
Zhurnal Eksperimental'noy i Teoreticheskoy Fiziki

Zh. Fiz. Khimii
Zhurnal Fizicheskoy Khimii

Zh. Neorgan. Khimii
Zhurnal Neorganicheskoy Khimii

Zh. Obshch. Khimii
Zhurnal Obshchey Khimii

Zh. Prikl. Khimii
Zhurnal Prikladnoy Khimii

Zh. Tekhn. Fiz.
Zhurnal Tekhnicheskoy Fiziki

Zh. Tekhn. Fiz.
Zhurnal Tekhnicheskoy Fiziki

Appendix B

PRINCIPAL SOVIET AUTHORS ON MASS TRANSFER RESEARCH AND DEVELOPMENTS

Author	No. of Refs. Cited	Institute	Specialty
<u>Absorption</u>			
Ganz, S. N.	8	Dnepropetrovsk Chemical Technology Institut imeni F. E. Dzerzhinskiy	Processes, equipment
Kafarov, V. V.	8	Moscow Institute of Chemical Technology imeni D. I. Mendeleev	Theory, calculation methods
Kishinevskiy, M. Kh.	17	Kishinev State University	Theory, rates
Kuz'minykh, I. N.	5	Moscow Institute of Chemical Technology imeni D. I. Mendeleev	Processes
Plit, I. G.	5	Dnepropetrovsk Chemical Technology Institut imeni F. E. Dzerzhinskiy	Processes
Pozin, M. Ye.	11	Leningrad Technological Institute imeni Lensovet	Theory, rates
<u>Adsorption</u>			
Avgul', N. N.	15	Moscow State University imeni M. V. Lomonsov	Calorimetry, Thermodynamics
Balandin, A. A.	15	Moscow State University imeni M. V. Lomonosov	Adsorption in catalysis
Bering, B. P.	15	Institute of Physical Chemistry, Academy of Sciences USSR	Thermodynamics
Bykov, V. T.	8	Far Eastern Affiliate imeni V. L. Komarov, Academy of Sciences USSR	Adsorbent structure (clays and earths)
Dubinin, M. M.	38	Sorption Processes Laboratory, Institute of Physical Chemistry, Academy of Sciences USSR	Adsorbent structure (carbon)

Author	No. of Refs. Cited	Institute	Specialty
Kiselev, A. V.	57	Moscow State University imeni M. V. Lomonosov	Adsorbent structure (silica gel), adsorbent mechanism
Lyashenko, V. I.	8	Institute of Physics, Academy of Sciences Ukrainian SSR	Chemisorption on semi- conductors
Neymark, I. Ye.	18	Institute of Physical Chemistry imeni L. V. Pisarzhevskiy, Academy of Sciences Ukrainian SSR	Adsorbent structure (silicates)
Rubinshteyn, A. M.	8	Institute of Organic Chemistry imeni N. D. Zelinskiy, Academy of Sciences USSR	Adsorption in catalysis
Serpinskiy, V. V.	14	Institute of Physical Chemistry, Academy of Sciences USSR	Thermodynamics
Shlygin, A. I.	8	Moscow State University imeni M. V. Lomonosov	Adsorption at electrodes
Timofeyev, D. P.	13	Institute of Physical Chemistry, Academy of Sciences USSR	Adsorption kinetics
Vol'kenshteyn, F. F.	13	Institute of Physical Chemistry, Academy of Sciences USSR	Chemisorption
Zaverina, Ye. D.	11	Institute of Physical Chemistry, Academy of Sciences USSR	Equilibria
Zhdanov, S. P.	10	Institute of Chemistry of Silicates, Academy of Sciences USSR	Adsorbent structure (Porous glasses)

Author	No. of Refs. Cited	Institute	Specialty
<u>Crystallization</u>			
Danilov, V. I.	12	deceased	Growth theory
Gorshteyn, G. I.	5	All-Union Institute of Chemical Reagents	Growth from solutions
Kamenetskaya, D. S.	4	Institute of Metal Studies and Metal Physics, Central Scientific Research Institute of Ferrous Metallurgy	Nucleation and growth
Kapustin, A. P.	6	Institute of Crystallography, Academy of Sciences USSR	Effects of ultrasonics
Lemmleyn, G. G.	4	Institute of Crystallography, Academy of Sciences USSR	Dendritic crystallization
Makarov, Ye. S.	7	Institute of Geochemistry and Analytical Chemistry imeni V. I. Vernadskiy, Academy of Sciences USSR	Crystal structure
Petrov, D. A.	6	Institute of Metallurgy imeni A. A. Baykov, Academy of Sciences USSR	Growth from melts
Pozdnyakov, P. G.	5	Central Scientific Research Laboratory of Piezoelectricity, Ministry of Electric Power Stations and Electrical Industry	Growth from solutions
Savitskiy, Ye. M.	4	Institute of Metallurgy imeni A. A. Baykov, Academy of Sciences USSR	Metal recrystalliza- tion
Sheftal', N. N.	7	Institute of Crystallography, Academy of Sciences USSR	Growth mechanism, single crystal techniques
Silant'yeva, N. I.	4	All-Union Institute of Chemical Reagents	Growth from solutions

Author	No. of Refs. Cited	Institute	Specialty
<u>Extraction</u>			
Alabyshev, A. F.	7	Leningrad Polytechnical Institute imeni M. I. Kalinin	Fused salt systems
Byk, S. Sh.	5	Scientific Research Institute of Synthetic Alcohols and Organic Products	Solubility by equilibria
Gel'perin, N. I.	7	Moscow Institute of Fine Chemical Technology imeni M. V. Lomonosov	Extraction equipment
Kafarov, V. V.	8	Moscow Institute of Chemical Technology imeni D. I. Mendeleev	Theory
Krupatkin, I. L.	20	Yaroslavl Technological Institut or Ivanovo Chemical Technology Institut	Liquid Phase Stability
Lantratov, M. F.	5	Leningrad Polytechnical Institut imeni M. I. Kalinin	Fused salt systems
Morachevskiy, A. G.	6	Leningrad State University imeni A. A. Zhdanov	Thermodynamics
Rozen, A. M.	5	Institute of Physical Problems imeni S. I. Vavilov	Uranium Extraction, Thermodynamics
Storonkin, A. V.	11	Leningrad State University imeni A. A. Zhdanov	Thermodynamics
Vdovenko, V. M.	15	Institute of Radium imeni V. G. Khlopin, Academy of Sciences, USSR	Uranium and fission product extraction; analytical application

Table

RECENT SOVIET STUDIES OF EXTRACTION EQUILIBRIA

<u>System</u>	<u>Temperature</u> (°C)
Benzene - formamide - acetone	25
Benzene - formamide - diethylformamide	25
Phenol - antipyrine - ligroine	25-120
Phenol - isopropylbenzene - methyl styrene	45
Water - acetic acid - benzene, toluene, xylene, diethyl ether, ethyl acetate, butyl acetate	20, 40, 60 and boiling
Water - aniline - aniline hydrochloride	25
Water - antipyrine - chloral hydrate	60-190
Water - ε - caprolactam - chloroform, dichloroethane, methylene chloride	--
Water - ethanol - benzene	35 - 65
Water - isopropanol - chloroethane	25
Water - isopropanol - chloroform, carbon tetrachloride	25
Water - methanol - dichloroethane	25
Water - methanol - hexane, heptane, octane, nonane	10, 20
Water - phenol - anthranilic acid	60 - 65
Water - phenol - antipyrine	25-120
Water - phenol, methyl ethyl ketone	20, 45, 75
Water - phenol, methyl styrene	20, 45, 70
Water - phenol - triethylamine	15, 35
Water - phenylene diamine - resorcinol	20-120
Water - pyridine - chloral hydrate	20-120
Water - quinoline - aniline	15-140
Water - quinoline - diethyl ether	15-140

Table 1 (cont.)

<u>System</u>		<u>Temperature</u> (°C)
Water	- salicylic acid - aminopyrine	60-170
Water	- salicylic acid - anthranilic acid	--
Water	- salicylic acid - gasoline	80-100
Water	- salicylic acid - phenol	25-100
Water	- salicylic acid - picric acid	--
Water	- dioxane - HCl, H ₂ SO ₄ , KCl, LiCl	--
Water	- ethanol - ammonium hydrogen fluoride	25
Water	- ethanol - sodium fluoride	0, 25
Water	- methanol - calcium chloride	15 - 25
Water	- hydrochloric acid - molybdenum chloride complexes - butanol, iso-butanol, iso-pentanol, butyl formate, ethyl acetate, diethyl oxalate	--
Water	- dibutyl ether - uranyl nitrate	25
Water	- diethyl ether - uranyl nitrate	15
Water	- nitric acid - plutonium, uranyl nitrates- tributyl phosphate	--
Water	- nitric acid - plutonium (IV) nitrate- tributyl phosphate (benzene, toluene, kerosene, freon, carbon tetrachloride)	--
Water	- nitric acid - plutonium (IV) nitrate - tributyl phosphate (benzene)	--
Water	- nitric acid - thorium nitrate - tributyl phosphate (benzene)	--
Water	- nitric acid - uranyl nitrate - tributyl phosphate (dibutyl ether, kerosene)	--

Table (cont.)

<u>System</u>	<u>Temperature</u>
<u>Molten Salt Systems:</u>	
$\text{SiO}_2 - \text{Al}_2\text{O}_3 - \text{MF}_2$ (M= Ba, Ca, Mg, Sr)	--
$\text{SiO}_2 - \text{MO} - \text{MF}_2$ (M= Ba, Mg, Sr)	--
$\text{SiO}_2 - \text{NaF} - \text{MF}_2$ (M= Ba, Ca, Mg, Sr)	--
$\text{SiO}_2 - \text{KF} - \text{CaF}_2, \text{MgF}_2$	--
$\text{SiO}_2 - \text{Na}_2\text{O} - \text{CaF}_2$	--
Tl Br - KNO_3 -AgCl, LiCl, NaCl, BeSO_4 , Na_2SO_4	--

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